United States Court of Appeals for the District of Columbia Circuit

TRANSCRIPT OF RECORD

No. 10,520

JOINT APPENDIX

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA CIRCUIT

PETER A. ASSEFF, and THE LUBRI-ZOL CORPORATION,
Appellants,

vs.

JOHN A. MARZALL, Commissioner of Patents,
Appellee.

227

Appeal from the United States District Court for the District of Columbia

United States Court of Appeals
For the
District of Columbia Circuit

FILED MAR 21 1950

Joseph W. Stewart
CLERK

WILSON - EPES PRINTING CO. - RE 6003 - WASHINGTON 1, D. C.

INDEX

PAGE

Complaint 2

Answer 10

Amendment to Answer 12

Opinion of Judge Tamm (District Court) 13

Judgment 22

Order Substituting Defendant 22

Notice of Appeal 23

Reporters Transcript of Testimony 23

Asseff Application—Exhibit 43 114
  File Wrapper 114
  Specification 114
  First Action 133
  First Amendment 134
  Supplemental Amendment 138
  Second Action 139
  Second Amendment 140
  Third Action 142
  Third Amendment 143
  Fourth Action 161
  Fourth Amendment 163
  Appeal to Board of Appeals 163
  Abandoned Cook Application 164
  Fifth Amendment 172
  Examiners report re Affidavit R. 75 173
  Decision of the Board of Appeals 175

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA CIRCUIT

No. 10,520

PETER A. ASSEFF, and THE LUBRI-ZOL CORPORATION,
Appellants,

vs.

JOHN A. MARZALL, Commissioner of Patents,
Appellee.

Appeal from the United States District Court for the District of Columbia

JOINT APPENDIX

Filed September 30 1947 Charles E. Stewart, Clerk

137 UNITED STATES DISTRICT COURT
FOR THE
DISTRICT OF COLUMBIA

PETER A. ASSEFF
Cleveland, Ohio; and
THE LUBRIZOL CORPORATION
Box 3057, Euclid Station
Cleveland, Ohio
Plaintiffs,
vs.
LAWRENCE C. KINGSLAND
Commissioner of Patents,
Defendant.

Civil Action No. 3967-'47

Complaint

To Issue Patent

The Plaintiff, Peter A. Asseff, is a resident of Cleveland, Ohio and is Chief Chemist in the laboratories of The Lubrizol Corporation located at Wickliffe, Ohio.

The Plaintiff, The Lubrizol Corporation, is a corporation organized and existing under and by virtue of the laws of the State of Ohio, with its principal office and place of business in the City of Wickliffe, State of Ohio.

I.

This Complaint is filed in accordance with the provisions of the Federal Laws of the United States as provided for under Revised Statute 4915.

II.

The Defendant, Lawrence C. Kingsland, is the Commissioner of Patents of the United States, an official resident of the District of Columbia, and is sued in his official capacity as Commissioner of Patents of the United States.

138 III.

Heretofore, to wit, prior to September 9, 1942, Peter A. Asseff, then a citizen of the United States and a resident of Cleveland, State of Ohio, was the original, first and sole inventor of a certain new and useful improvement in LUBRICATING COMPOSITION; and said invention was a new and useful improvement not known or used by others in this country before his invention thereof, and not patented or described in any publication in this or any foreign country before his invention thereof or more than one year prior to his said application for Letters Patent therefor, nor at public use or on sale in this country, for more than one year prior to his said application, nor ever abandoned; nor ever made the subject of any foreign application filed by him or his legal representatives prior to his said application; and said Peter A. Asseff, did on September 9, 1942, duly file in the Patent Office of the United States an application in writing praying for the issuance to him of Letters Patent of the United States for said invention.

IV.

The said application has been passed upon by the Commissioner of Patents in the person of the Primary Examiner who allowed no claims and specifically refused to allow the following claims:—

Claim 1: A lubricating composition containing a characterizing amount of the product obtained by the simultaneous admixture of capryl alcohol, phosphorus pentasulphide, and zinc oxide.

Claim 5: A lubricating composition for Diesel engines capable of inhibiting ring sticking and corrosion of the bearing surfaces of the engine with which the same comes in contact, especially when such bearing surfaces comprise a characterizing amount of a metal of the class consisting of nickel, copper, silver, cadmium, lead and tin, comprising a major proportion of highly refined solvent extracted mineral lubricating oil having a viscosity index of the Dean and Davis scale of over 75 and a minor amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide.

139 Claim 9: A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a separate detergent of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur.

Claim 10: A lubricating composition containing a characterizing amount of the product obtaining by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a metal-containing detergent of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur.

Claim 11: A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a calcium salt of a petroleum sulphonic acid.

Claim 12: An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a separate detergent of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur.

Claim 13: An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and a metal-containing detergent of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur.

Claim 14: An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a calcium salt of a petroleum sulphonic acid.

Claim 15: A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and from about .1% to about 20% of a stable oil soluble halogen bearing organic compound having a vapor pressure less than atmospheric at 140° C.

140 Claim 16: A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a separate oil-soluble organic corrosion inhibitor.

Claim 17: A lubricating composition containing a characterizing amount of the product obtained by the simultaneous admixture of capryl alcohol, phosphorus pentasulphide, and zinc oxide and a separate detergent of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur, and from about .1% to about 20% of a stable oil-soluble halogen-bearing organic compound having a vapor pressure less than atmospheric at 140° C.

Claim 18: A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, a separate detergent of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur, and a separate oil-soluble organic corrosion inhibitor.

Claim 19: An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phophorus pentasulphide, a separate detergent, of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur, and from about .1% to about 20% of a stable oil-soluble halogen-bearing organic compound having a vapor pressure less than atmospheric at 140° C.

Claim 20: An addition agent capable of inhibiting ring sticking and corrosion for lurbicating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, a separate detergent of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur, and an oil-soluble organic corrosion inhibitor.

Claim 21: A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, a separate detergent, of the class consisting of oil-soluble organic compounds, containing a metal atom directly attached to an element of the

class consisting of oxygen and sulphur, from about
141 .1% to about 20% of a stable oil-soluble halogen-bearing organic compound having a vapor pressure less than atmospheric at 140° C., and an oil-soluble organic corrosion inhibitor.

Claim 22: A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and an oil-soluble metallic phenate.

Claim 23: A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and an oil-soluble metallic phenate derived from an ester of salicylic acid.

Claim 24: A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and the magnesium phenate derived from lauryl salicylate.

V.

The grounds upon which the Primary Examiner rejected said claims were:—

(1) That claims 1, 5 and 9 thru 24 lacked invention over the patent to Cook 2,344,392.

(2) That claims 9 thru 24 lacked invention over Cook and Salzberg 2,063,629 patents in view of the patents to Wilson 2,280,419 and McNab 2,289,795.

(3) That claims 16, 18, 20 and 21 were too broad and indefinite in claiming broadly "corrosion inhibitors".

VI.

The applicant thereupon duly appealed from the decision of the Primary Examiner to the Board of Appeals who affirmed the rejection of the Primary Examiner by its decision dated March 31, 1947.

VII.

The Board of Appeals in considering this case

(1) affirmed the rejection of claims 1, 5 and 9 thru 24 as lacking invention over the patent to Cook.

(2) affirmed the rejection of claims 16, 18, 20 and 21 as too broad and indefinite with respect to "corrison inhibitors".

142 (3) affirmed the invention of the claims on the patents to Salzberg and Cook in view of the patents to McNab and Wilson.

VIII.

The Commissioner of Patents has refused to allow any of the claims on the ground that they are anticipated by the aforesaid prior art.

IX.

The plaintiff disagree with the foregoing contentions of the Commissioner of Patents and contend that the claims are patentable over said prior art.

X.

No appeal has been taken from the decision of the Board of Appeals to the United States Court of Customs and Patent Appeals; and no such appeal is pending or has been decided, and this Bill is filed within six months of the date of refusal by the Board of Appeals to allow the said application of the said Peter A. Asseff, Serial No. 457,746.

XI.

The aforesaid application of Peter A. Asseff, Serial No. 457,746, the invention covered thereby, and the patent to issue thereon, was, by instrument in writing, duly assigned and transferred to the Lubrizol Corporation, an Ohio corporation, plaintiff herein; said assignment was duly recorded in the United States Patent Office at Liber

Q-192, page 218 of the Transfers of Patents, and plaintiff corporation is now the sole and exclusive owner of the entire right, title and interest in and to said application and the invention and discovery disclosed and claimed therein. The assignment aforesaid or a certified copy thereof is ready in Court to be produced and profert thereof is hereby made.

143 XII.

The Commissioner of Patents has refused and still refuses to grant Letters Patent upon the aforesaid application, Serial No. 457,746, and particularly claims 1, 5 and 9 thru 24 inclusive to which plaintiffs are lawfully entitled.

WHEREFORE, plaintiffs bring this Bill under and in accordance with the Statutes in such cases made and provided and respectfully prays:

(a) That the Court adjudge and decree that Peter A. Asseff is the first, true and original inventor of the improvements set forth in the claims in said application, Serial No. 457,746.

(b) That the Court adjudge and decree that Plaintiff, THE LUBRIZOL CORPORATION, is entitled according to law to receive Letters Patent of the United States for the aforesaid invention and discovery of the said Peter A. Asseff, as specified and pointed out and distinctly claimed in said application, Serial No. 457,746, as the assignee of said Peter A. Asseff.

(c) That the Court authorize and direct the Commissioner of Patents to issue Letters Patent upon said application Serial No. 457,746 containing the claims thereof and any other claims which this Court may adjudge lawful and proper.

(d) To grant to plaintiffs such other and further relief as equity may require and which to the Court may seem meet and proper.

144 (e) To issue a subpoena ad respondum directed to Lawrence C. Kingsland, Commissioner of Pat-

ents of the United States under the Seal of this Honorable Court requiring him to appeal and answer this Bill of Complaint and Abide and Further Order of this Court.

PETER A. ASSEFF
THE LUBRIZOL CORPORATION
By Oberlin & Limbach
And Oscar C. Limbach
Their Attorneys

/s/ Almon S. Nelson
Almon S. Nelson
1051 Earle Bldg.
Washington, D. C.
Na 6060

* * * *

145 Filed Oct 28 1947 Harry M. Hull, Clerk

Answer to the Complaint.

To the Honorable the Justices of the District Court of the United States for the District of Columbia.

Defendant admits the allegations of the first two unnumbered paragraphs.

I, II. He admits the allegations of paragraphs I and II.

III. He admits that on September 9, 1942, plaintiff Peter A. Asseff, claiming to be the original, first and sole inventor of certain alleged new and useful improvements in "Lubricating Composition", filed in the Patent Office an application for patent thereon, to which application Serial No. 457,746 was given, and in said application made averments corresponding to the allegations of paragraph III. He denies, for reasons hereinafter given, that said allegations are sufficient to justify the issuance of a patent on said application.

146 IV, V, VI, VII, VIII. He admits the allegations of paragraphs IV to VIII, inclusive.

IX. He admits that plaintiffs disagree with the decision of the Board of Appeals affirming the rejection of the primary examiner of claims 1, 5, and 9 to 24, inclusive, of said application, which claims are as set out in paragraph IV. Defendant denies plaintiffs' contention that the said claims are patentable over the prior art.

X. He admits the allegations of paragraph X.

XI. He admits the allegation of assignment of said application of Peter A. Asseff to plaintiff, The Lubrizol Corporation, but denies that the subject-matter claimed in said application constitutes a patentable invention. He admits that plaintiffs should produce in Court a certified copy of said application.

XII. He admits that he has refused and still refuses to issue a patent on said application containing any of said claims 1, 5, and 9 to 24, inclusive. He denies that plaintiffs are lawfully entitled thereto. He states that the said claims are unpatentable for the reason that they are vague and indefinite to the extent that they fail to meet the requirements of Section 4888 R. S. (U. S. C., title 35, sec. 33) that an applicant for patent shall point out and distinctly claim the part, improvement, or combination which he claims as his invention or discovery, for the reason that the said claims recite nothing amounting to an invention over the following prior patents and for the reasons given by the Examiner in his statements dated October 16, 1945, January 27, 1947, and March 5, 1947, and by the Board of Appeals in its decision dated March 31, 1947:

147

Salzberg et al.,	2,063,629,	Dec. 8, 1936,
Wilson,	2,280,419,	April 21, 1942,
McNab,	2,289,795,	July 14, 1942,
Cook et al.,	2,344,392,	March 14, 1944.

Profert of copies of these patents is hereby made.

/s/ W. W. Cochran
Solicitor, U. S. Patent Office,
Attorney for Defendant.

* * * *

148 Filed Oct 30, 1947 Harry M. Hull, Clerk

Amendment to the Answer to the Complaint.

To the Honorable the Justices of the District Court of the United States for the District of Columbia.

Under the provisions of Rule 15(a) of the Federal Rules of Civil Procedure Defendant hereby amends Paragraph XII of the ANSWER TO THE COMPLAINT to read as follows:

XII. He admits that he has refused and still refuses to issue a patent on said application containing any of said claims 1, 5, and 9 to 24, inclusive. He denies that plaintiffs are lawfully entitled thereto. He states that the said claims are unpatentable for the reason that they are vague and indefinite to the extent that they fail to meet the requirements of Section 4888 R. S. (U. S. C., title 35, sec. 33) that an applicant for patent shall point out and distinctly claim the part, improvement, or combination which he claims as his invention or discovery, for the reason that the said claims recite nothing amounting to an invention over the following prior patents and for the reasons given by the Examiner in his statements dated October 16, 1945, January 27, 1947, and March 5, 1947, and by the Board of Appeals in its decision dated March 31, 1947:

149

Salzberg et al.,	2,063,629,	Dec. 8, 1936,
Wilson,	2,280,419,	April 21, 1942,
McNab,	2,289,795,	July 14, 1942,
Cook et al.,	2,344,392,	March 14, 1944,
Musselman,	2,419,325,	April 22, 1947,
Asseff,	2,261,047,	Oct. 28, 1941.

Profert of copies of these patents is hereby made.

/s/ W. W. Cochran
Solicitor, U. S. Patent Office,
Attorney for Defendant.

* * * *

150 Filed Oct 3 1949 Harry M. Hull, Clerk

Memorandum Opinion

In this action, brought under the provisions of §4915 of the Revised Statutes (35 U. S. C. 63), plaintiff Peter A. Asseff, as the original applicant, and the Lubrizol Corporation, as assignee of Asseff's right, title and interest in the said application, seek to have this Court order the defendant, the Commissioner of Patents, to issue a patent upon certain claims of the Peter Asseff application. Peter Asseff's application, Serial #457,746, was filed in the Patent Office on September 9, 1942. The claims of the patent application upon which plaintiffs base this action are claims numbered one, five, and nine to twenty-four inclusive. The specification of the application describes the invention as one relating to lubricating compositions
151 which are particularly suited for use under high
temperature conditions such as those encountered in internal combustion engines of the Diesel type. It appears that ordinary lubricants utilized under the above-described conditions cause deposits to form, resulting in ring sticking and bearing corrosion. In other words, plain lubricating oil is generally incapable of use by itself under vigorous operating conditions such as high temperatures. Ordinary lubricating oil, no matter how highly refined, deteriorates by oxidation or decomposition, and as a result of this deterioration either fails to provide adequate lubrication, or the by-products of such deterioration collect as deposits which interfere with the operation of the equipment being lubricated and, or, the operation of the oil as a lubricant. The plaintiffs' composition is claimed to consist of a chemical addition agent which eliminates the above-described disadvantages of ordinary lubricants. To accomplish these objectives the plaintiffs' patent application relates to the compounding of a lubricating com-

position in which the main or base constituent is a hydrocarbon oil such as mineral lubricating oil, and in which the minor constituent is a compound, the molecular structure of which includes the combination of zinc, the capryl alcohol radical and the di-thiophosphate radicle. Other compounds may be included in the composition such as detergents, pour point depressors, and extreme pressure agents to meet particular problems of lubrication.

The plaintiffs' application was initially rejected in the Patent Office by the primary examiner on the grounds that (1) the eighteen claims before the Court lacked invention over a patent issued to Cook, et al. (Patent No. 2,344,392;); that claims nine through twenty-four lacked invention over Cook and Salzberg patents (#2,063,629) in view of the patents to Wilson (#2,280,419) and McNab (#2,289,795); (3) that claims sixteen, eighteen, twenty and twenty-one were too broad and indefinite in claiming "corrosion inhibitors". The Board of Appeals affirmed the decision of the primary examiner by a decision
152 dated March 31, 1947. The defendant has, according to the complaint, refused to allow any of plaintiffs' claims on the ground that they are anticipated by the prior art, and the plaintiffs initiated this action within the statutory period seeking to compel the issuance of the patent sought by plaintiffs.

The plaintiffs' claims, numbering eighteen, as set forth in their complaint, all define a composition of matter designated either a lubricating composition (as in Claim one) or an addition agent (as in Claim twelve). All of the claims are distinguished by the inclusion therein of the "product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulfide", and it is this "product" upon which the plaintiffs base their contention of patentable novelty in the claims. While certain of the plaintiffs' claims (such as nine, ten and eleven) call for an additional compound as a detergent, and others (such as claims fifteen and sixteen) call for an additional compound as a "corrosion inhibitor", all claims in this action are based upon "preparing the zinc salt of the reaction product of capryl alcohol with phophorus pentasulfide".

The Prior Art

On December 8, 1936, a patent (#2,063,629) was granted to Paul L. Salzberg, et al., for a compound employed as "corrosion inhibitors * * * in lubricants for bearings which operate under high pressure". (p. 3, col. 2, lines 41-43) The Salzberg patent relates to the production of acid esters and salts of dithiophosphorus acid. Zinc is mentioned as one of the metals forming the salt (p. 3, col. 2, line 19) and the ester portion is provided by aliphatic alcohols containing at least ten carbon atoms (p. 1, col. 1, lines 33-36).

On July 14, 1942 a patent was issued to John G. McNab on an application filed September 1, 1939 for compositions related to "improved lubricants and especially crank case oils for the lubrication of internal combustion engines and particularly for high pressure fuel injection
153 engines such as Diesel engines". The McNab patent discloses the use of a salt of a dithiophosphoric acid ester, especially calcium di-octyl dithiophosphate (p. 3, col. 2, line 70) in conjunction with metal soaps of natural and synthetic fatty acids and naphthenic acid (p. 2, col. 2, lines 56-61). The McNab patent shows the use of a salt of dithiophosphate ester in which the alkyl radicle contains eight carbon atoms (p. 3, col. 2, line 70). The McNab patent teaches that viscosity improving agents and pour point depressors may also be included for their particular function (p. 4, col. 2, lines 17-30).

On April 21, 1942, a patent was granted to Chester E. Wilson (#2,280,419) covering addition of constituents to mineral lubricating oils to give them special characteristics especially adapting them to severe service uses "such as are encountered in Diesel engines" (p. 1, col. 1, lines 1-6).

The Wilson patent describes a lubricant containing an oil soluble salt of the so-called "Mahogany" sulfonic acids, including the zinc salt, together with oil soluble metal salts of theophenals (p. 1, col. 1, lines 37-45). The sulfonic acids are said to function as detergents, while the sulfur of the theophenol imports anti-corrosive properties (p. 4, col. 1, lines 35-43).

On March 14, 1944, a patent was granted to Elmer W. Cook, et al. (#2,344,392) on an application filed November 8, 1941, as a continuation in part of an application filed July 11, 1941. This patent describes the preparation of heavy metal salts of the dicapryl ester of dithiophosphoric acid, and teaches that such salts are useful as sludge preventers and anti-corrosive agents for lubricating oils used in internal combustion engines (p. 1, col. 1, lines 8-11 and 42-47). Zinc is specifically mentioned as one of the heavy metals (p. 1, col. 1, lines 1-23). These heavy metal salts are stated to cooperate with the heavy metal salts of diamyl phenol sulfides described in Cook, et al.'s patent (#2,299,626), as well as with alkaline earth metal salts of petroleum sulfonates (p. 1, col. 2, lines 48-53). The dithiophposphoric acid ester salts are described as
154 being good detergents and to possess important non-
oxidizing or anti-oxidant properties (p. 1, col. 1, lines 43-48). The conjoint use of the reaction product with other detergents and corrosion inhibitors is also taught by this patent.

On October 28, 1941 a patent was issued to Peter A. Asseff (#2,261,047), one of the plaintiffs herein, on an application filed on July 28, 1941, covering a compounded lubricant. The patent describes a zinc methyl cyclohexanol dithiophosphate salt as an additive for lubricating oil, and discloses that metal phenates may be used in conjunction therewith (p. 2, col. 1, lines 6-12).

It will be observed that the claims here in issue relate in all instances to the use of a capryl alcohol, which alcohol contains only eight carbon atoms. Plaintiffs claim the use of capryl alcohol distinguishes their claims from Salzberg and McNab as outlined above. Salzberg utilized an aliphatic alcohol, but plaintiffs' witness, Dr. Smith, testified that capryl alcohol is an aliphatic alcohol. McNab (p. 3, col. 2, line 70) utilized an octyl alcohol and Dr. Smith testified that capryl alcohol is an octyl alcohol. (Tr. p. 103).

Long prior to plaintiffs' application for the patent here in suit, the art was conversant with the use of dioctyl dithiophosphate salt as indicated by the Salzberg and McNab patents heretofore briefly described. The alcohol used by plaintiffs falls clearly within the disclosure of the prior art. The preparation and use of the capryl ester was a logical and obvious adaptation of the teaching of the prior art, requiring for its accomplishment only the skill of the art. It is further noted that Salzberg indicates the equivalency of the calcium and zinc salts (p. 3, col. 2, lines 17-20) by specific reference to each, and it follows that plaintiffs' preparation of the zinc salt is plainly foreshadowed by the teaching of the prior art. The method of making the specific ester and salt therefrom were, of course, available to plaintiffs. Their production and use were no more than would be reasonably expected from one skilled in the art and did not constitute invention. *Sonneborn Sons* v. *Coe,* 70 App. D. C. 97, 104 F. 2d 230;
155 *Rembert, et al.* v. *Coe,* 78 U. S. App. D. C. 51, 136 F 2d 793.

The Wilson patent described briefly above shows that the advantageous use of plural additives, and the addition of halogen containing compounds, metal alcoholates and other compounds for their particular functions was recognized in the art. The plaintiff Asseff was following such prior art when he added a separate corrosion inhibitor, or a separate detergent, to dicapryl dithiophosphate salt. Applying these facts to plaintiffs' claims nine to twenty-four, the Court finds these claims lack invention and patentability over Salzberg, McNab and Wilson.

Claim one of plaintiffs' claims set forth a "lubricating composition containing a characterizing amount of the product obtained by the *simultaneous admixture* of capryl alcohol, phosphorus pentasulfide and zinc oxide" (emphasis supplied). Plaintiffs base strong contention for their claim of patentability over the Cook, et al., patent, on the grounds of reduction to practice prior to the filing of the Cook patent. Evidence was introduced by plaintiffs in an effort to establish this prior reduction to practice. Insofar as Claim One is concerned, the plaintiffs' witness, Asseff, testified (Trans. pp. 67 and 68) that no exhibit offered by the plaintiffs related to any "simultaneous admixture" of zinc, capryl alcohol and phosphorus materials. Insofar as this claim is concerned, plaintiffs are therefore remitted to the filing date of their application, which date being subsequent to the filing date of the Cook patent establishes the Cook patent as prior art to plaintiffs' claim. *Alexander Milburn Co.* v. *David-Bourbonville Company,* 270 U. S. 390; *Minnesota Mining & Manufacturing Co.* v. *Coe,* 69 App. D. C. 256, 100 F. 2d 429. The Court accordingly finds for the defendant upon this claim.

Claim Five of plaintiff's application is as follows:

"A lubricating composition for Diesel engines capable of inhibiting ring sticking and corrosion of the bearing surfaces of the engine with which the same comes in contact, especially when such bearing surfaces comprise a characterizing amount of a metal of the class consisting
156 of nickel, copper, silver, cadmium, lead and tin,
comprising major proportion of highly refined solvent extracted mineral lubricating oil having a viscosity index of the Dean and Davis scale of over 75 and a minor amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide."

The plaintiffs in support of their contention as to the patentability of this claim take the position that their work ante-dates the Cook application, admitting that the

Cook patent discloses the subject matter of plaintiffs' claims. (Defendant's brief, p. 19). As pointed out heretofore, plaintiffs' application here in action was filed on September 9, 1942. Cook, et al., it will be recalled, filed their application,—which matured into patent #2,344,392—on November 8, 1941, as a continuation of an application filed July 11, 1941.

Plaintiff Asseff in his testimony identified Plaintiffs' Exhibit #23 as establishing the claims here in suit, referring specifically to the Code number 601-302 as first showing the preparation of the zinc capryl alcohol dithiophosphate. The plaintiffs' witness in answer to a question stated that this is the first time at which that particular compound was made. (Trans. pp. 66 and 67). An examination of this exhibit discloses that this compound was first prepared on September 11, 1941 and tests started and completed between that date and October 1, 1941. It is observed from plaintiffs' brief (p. 21) that the statement is made with reference to the "use in a lubricant of the zinc salt of the acidic reaction product of capryl alcohol and P_2S_5 that "such invention was completed by Asseff with his engine tests completed October 3, 1941."

It has been established that the Cook application filed on November 8, 1941, was a continuation of the application filed on July 11, 1941. It so states on its face. (p. 2, col. 2, lines 29-31). Legally, therefore, the effective date of the Cook patent is July 11, 1941 since the two applications cover the same subject matter. *Godfrey* v. *Eames,* 68 U. S. 317; *Smith* v. *Goodyear Dental Vulcanite Co.,* 93 U. S. 486. That the parent application's date is carried over to the continuing application or patent
157 is well established *Hunt* v. *Mallinckrodt Chemical Works,* 72 F. Supp. 865; *In Re Lotz,* 57 App. D. C. 402, 17 Fed. 2d 1008. Plaintiffs have therefore not only failed to carry the burden of proof to establish prior reduction to practice but their evidence, in the light of the above, requires a finding for the defendant unless plaintiffs' claim

is not covered by the Cook applications. An examination of the Cook parent application (P's. Ex. 43) discloses that it embraces all the components of the reaction product defined in the plaintiffs' claims even to the preference for zinc as a heavy metal, that the actyl alcohol radicle is the preferred alkyl radicle and that capryl alcohol should be used. It follows that plaintiffs' claims, and particularly Claim Five, are unpatentable over Cook.

One further composition was described by plaintiff Asseff as a composition falling within the terms of Claims One and Five. This compound identified as Code number 601-310 is included in plaintiffs' Exhibit #24. The compound was made on September 18, 1941 and was "observed for short periods of storage" but no engine tests were ever run on this compound (Trans. pp. 57 and 69). While the case of *Knutson* v. *Gallsworthy,* 82 U. S. App. D. C. 304, 164 F. 2d 497, raises a question as to whether this action constituted a "reduction to practice", it is unnecessary to decide this point since the preparation itself was more than two months subsequent to the filing of the parent Cook application, and was, accordingly, covered by the prior disclosure of Cook as set out heretofore.

It appearing from the facts and findings set forth to this point that plaintiffs have failed to prove priority over Cook by establishing plaintiffs' Claims One and Five, it remains to consider the defendant's rejection of plaintiffs' Claims Nine through Twenty-four in the light of the Cook patent.

Plaintiffs' Claims nine, ten and eleven define the so-called zinc dicapryl dithiophosphate compound plus a separate detergent of the class containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur. In Claim eleven the compound
158 is described as a calcium salt of a petroleum sulfonic acid. It will be noted, however, that the Cook, et al. patent teaches that the dithiophosphates may be used in combination with detergents. The heavy metal salts of

diamylphenol sulfide disclosed in the Cook patent (p. 1, col. 2, line 49) respond to the definition of the separate detergent set out in plaintiffs' claims nine and ten and since that same material is disclosed in the parent Cook application, that disclosure is prior art insofar as plaintiffs are concerned. Relative to plaintiffs' claim eleven, calcium petroleum sulfonate used as a detergent is old in the Wilson patent (p. 1, col. 2, line 10) and therefore the use of that compound with the dicapryl dithiophosphate of Cook is within the skill of the art.

Plaintiffs' claims fifteen and sixteen define the basic compound of claim one, heretofore set out on several occasions, plus a "separate oil-soluble organic corrosion inhibitor" (claim sixteen) defined in claim fifteen as "halogen bearing". Halogen bearing compounds are shown in Wilson (p. 6, col. 1, line 1) and the use of such compounds admixed as stated by Cook is non-patentable.

Plaintiffs' claims seventeen to twenty-one describe the basic product,—"the zinc dicapryl dithiophosphate compound" combined with the detergent of claim nine and the corrosion inhibitor of claim sixteen. However, it will be recalled that the Wilson patent teaches those skilled in the art to utilize a plurality of additive agents and the parent Cook, et al. application disclosed that the specific heavy metal salts of the dithiophosphoric esters disclosed therein should be used with other materials. There is, therefore, nothing inventive in these claims.

Plaintiffs' claims twenty-two, twenty-three and twenty-four, defining species of compositions under claim nine, include as the separate detergent a "metallic phenats" (claims twenty-two and twenty-three), or "magnesium phenate" (claim twenty-four). Phenates as additive agents are old in both the McNab (called "phe-
159 nolates" therein) and Wilson patents. The inclusion of this material, old in the art, with the basic compound of the Cook patent is not new or inventive.

The findings of the Court having established that the plaintiffs' claims fail to ante-date the anticipatory effect of the Cook, et al. patent, which anticipates all of the claims of the plaintiffs herein; and that plaintiffs' claims nine through twenty-four lack invention and patentability over McNab, Salzberg and Wilson, it is unnecessary for the Court to pass upon the defendant's contention concerning the plaintiffs' failure to meet statutory requirements relating to particularity and distinctness.

Judgment for the defendant. Counsel will submit the necessary Order.

/s/ Edward A. Tamm
Judge

Dated:
October 1st 1949.

* * * *

160 Filed Oct 17 1949 Harry M. Hull, Clerk

Judgment

This action came on to be heard at this term and thereupon, upon consideration thereof, it is this 17th day of October, 1949

ADJUDGED that the complaint be and it is hereby dismissed, with costs against the plaintiffs.

The memorandum opinion of the Court dated October 1, 1949 shall be considered findings of fact and conclusions of law.

/s/ Edward A. Tamm
Judge.

* * * *

161 Filed Dec 16 1949 Harry M. Hull, Clerk

Order.

Upon consideration of the fact that John A. Marzall succeeded to the office of Commissioner of Patents on December 1, 1949, in place of Lawrence C. Kingsland, who resigned, and it appearing that there is substantial need for continuing and maintaining the above-entitled action, now therefore it is this 16th day of December 1949

ORDERED that John A. Marzall, Commissioner of Patents be and he is hereby substituted as party defendant in place of Lawrence C. Kingsland, formerly Commissioner of Patents.

H. A. Schweinhaut,
Judge.

* * * *

162 Filed Dec 16 1949 Harry M. Hull, Clerk

Notice of Appeal

Notice is hereby given that Peter A. Asseff and The Lubri-Zol Corporation, Plaintiffs herein, hereby appeal to the United States Court of Appeals for the District of Columbia from the final judgment entered in this action on October 17, 1949, dismissing Plaintiff's Complaint.

/s/ Almon S. Nelson
Almon S. Nelson
Attorney for Plaintiffs

* * * *

2 *Proceedings*

THE DEPUTY CLERK OF THE COURT: Asseff, et al., vs. Kingsland. Mr. Nelson and Mr. Cochran.

MR. SCHIMMEL: It is Mr. Schimmel instead of Mr. Cochran.

THE COURT: Are both sides ready?

MR. NELSON: Yes, Your Honor.

THE COURT: Will counsel identify themselves to the reporter, please?

MR. NELSON: I have already done that, and I think he knows Mr. Limbach.

Your Honor, may I present Mr. Limbach of the Ohio Bar, who would like to present the case for the plaintiffs.

THE COURT: It is a pleasure to have you here.

MR. LIMBACH: Thank you, Your Honor.

MR. SCHIMMEL: I am Joseph Schimmel, representing the Commissioner of Patents.

MR. LIMBACH: May it please Your Honor, the plaintiffs in this case are the Lubrizol Corporation and Peter A. Asseff, the applicant and assignor to the Lubrizol Corporation of his application for patent filed September 9, 1942, Serial No. 457,746, for a patent on an invention relating to lubricants.

Before going into a discussion of the issue, being the claims which were refused by the Patent Office, I think it might be well to say a word about the application
3 so that you might have a picture of the type of business that the Lubrizol Corporation is engaged upon.

The Lubrizol Corporation is a company in Cleveland whose primary business is the manufacture and sale of chemicals that are used primarily as addition agents to lubricating oils, gasolines, and similar products.

These addition agents are added to lubricating oils and gasolines for a variety of reasons, that is, for the purpose of improving the base material of the oil or gasoline in a variety of ways.

Lubricating oils and gasolines deteriorate as by oxidization, by coming in contact with oxygen as it is present in the air, either from storage, or more particularly from use as, for example, in the engine of an automobile, or in the rear axle of an automobile, where the lubricant is churned up and comes in contact with air or at high temperatures it oxidizes and deteriorates.

Deterioration power of various of the lubricants varies in a lot of ways. I do not think it is necessary to go into the details of the other ways of deterioration, oxidation being the illustration of one of the many things that happen.

In addition to the use of those chemicals or addition
agents for the purpose of preventing deterioration, they
are added for the purpose of imparting properties to the
lubricant which the basic composition does not have.
4 One of those properties is ability of the particular
lubricant to withstand high loads, or as we com-
monly refer to that property in the industry as "extreme
pressure." That is encountered wherever you have lubri-
cants between bearing surfaces that operate under high
pressure, where pressures could be, for example, well
above 10,000 pounds per square inch, and plain mineral
oil would be squeezed out and would be completely dis-
placed finally and be unable to function as a proper lubri-
cant without the help of these chemical addition agents
that are used.

These conditions that are encountered by a lubricant in use are attended by a deterioration of the lubricant, that being the reason why we have to change the oil in our automobile crankcase, in our gear cases, and rear axles. That deterioration comes by a breakdown of the oil, or resulting from a breakdown of the oil, and builds up in the oil itself bodies which are harmful not only to the lubricant film, not only to the proper operation of the lubricant film, but the decomposition product builds up as deposit on the surfaces that are to be protected, and not only there but also in places where the oil is required to circulate, and blocking the circulation of the oil and generally interfering with the free action of either the parts being lubricated or the movement of the oil body itself.

The business of the Lubrizol Corporation is that
5 of a supplier to the petroleum industry of these
chemicals. At the present time a very large pro-
portion of all of the lubricants that are sold and used are
fortified with one or more of these chemicals.

The particular claim or head claim that is here on appeal—well, they are not on appeal but in this action they are de novo—identifies the composition as a lubricating composition containing a characterizing amount of the product obtained by the simultaneous admixture of capryl alcohol, phosphorus pentasulphide, and zinc oxide.

That claim is a bit narrower than some others since other claims that do not require that these three components named in Claim 1 be simultaneously admixed, and the chemical reaction can be carried on several ways, but essentially the compound we are talking about is a zinc salt produced by first reacting capryl alcohol with phosphorus pentasulphide, and the product is the zinc salt which is then produced.

Some of the other claims in the case, for example, Claim 10 calls for the additional presence in the lubricant of a detergent, and that component is precisely what its name signifies and operates precisely in the manner that its name signifies, namely, a chemical compound put in the oil for purpose of keeping the parts clean.

Then there are other claims, such as for example 15, which are further specific in requiring the addi-
6 tional presence in the composition, in addition to the first named component, of a halogen bearing organic compound. That compound is added, as clearly stated in the specification, for the purpose of increasing the film strength of this lubricant with which we are concerned.

Then there are other claims, for example, Claim 20, which calls for the additional presence and still in the same basic composition of an additional corrosion inhibitor.

And then in the claims, such as 23 and 24, the detergent material in the composition is specifically identified, first as a metallic phenate and secondly, and more specifically as magnesium phenate.

These claims have been rejected by the Patent Office on prior art, which is listed in the answer, not all of which, however, are relied upon by the Board of Patent Appeals in its decision.

In the amended answer, Your Honor, or in the original answer, four patents were listed: Salzberg, Wilson, McNab, and Cook. They are the patents which were relied upon by the Board of Appeals.

In the amended answer, counsel for the Commissioner have added two additional patents, Musselman and the plaintiff Asseff's own prior patent, and the reasons for the addition of these patents will presumably become apparent as Mr. Schimmel presents his case.

7 At the present time I have no idea why he cited them, because one is the plaintiff's own prior patent, and the effective date of the other of which is after the date of the present case.

The principal issues here are, first, the anticipatory nature of these patents, which are earlier in that they clearly antedate the work of this patent. Representative of these patents was the Salzberg patent, and that is about the only one—Salzberg and McNab—no, Salzberg is the only one that is clearly in that category.

The second group of patents represented by the patent of Cook was alleged to be antedated by the work this applicant did, evidence of which we will present during the trial of this case.

During the proceedings in the Patent Office, and the final rejection of the case by the primary examiner, we filed simultaneously with our brief in the Board of Appeals an affidavit of the applicant, corroborating, as we thought properly, and in which affidavit we put in certain evidence by which we sought to show that the applicant had completed his invention prior to the effective date of the principal Cook patent.

When that affidavit, under Rule 75, was filed with the Board of Appeals, the case was remanded to the primary examiner, who refused to enter the amendment on the ground that there was not made a sufficient showing
8 explaining the delay or lateness in filing the affidavit.

In response to that I filed an affidavit explaining the reasons why, and I say that the affidavit was timely in view of the prosecution of the application and the manner in which the patent examiner had not applied the references, and that was taken to the Commissioner on petition, who affirmed the primary examiner in his refusal to admit the affidavit.

But these proceedings, being a trial de novo of the entire issue, makes it possible to bring in that evidence to show that the applicant Asseff was the first inventor of the claim.

The case will be presented in two parts, Your Honor, the first part which will pertain to the early patents, to show that they did not aniteipate this invention, and that there is no teaching in them or disclosure of the present invention. And secondly, with respect to certain art, such as the Cook patent, which does contain a disclosure but no claim on this invention, and the present applicant made his invention prior to the effective date of it.

MR. SCHIMMEL: If it please the Court, this is an action under Section 4915 of the Revised Statutes. The applicant here was dissatisfied with the Board of Appeals decision which affirmed the primary examiner's report or rejection of all the claims of his application as unpatentable.

As you have heard, the invention is of a lubricating
composition, and it is particularly directed to the
9 incorporation in ordinary lubricating oil, whether
of ordinary grade or highly refined grade, a particular material which is supposed to impart certain characteristics to the oil.

As the applicant in his own specification stated: The invention comprises the provision of lubricating compositions which contain a characterizing amount of the compound, the molecular structure of which includes a combination of zinc, capryl alcohol radical, and di-thio phosphate radical, and he alleges that particular material imparts non-corrosive characteristics to the lubricating composition.

There are 18 claims set forth in the bill of complaint, and through each one of these claims runs this thread of the use of this particular compound as the novel invention and patentable element of all the claims.

There are additional agents added but the evidence will show that each of the agents are old in the art, and the novelty is directed to this particular zinc, capryl alcohol and di-thio phosphate radical compound.

In the Patent Office the Examiner found that this applicant here was not the first to use that particular compound in a lubricating oil composition, and if Your Honor will refer to the folder on your desk, B of that folder is the patent disclosure, Cook and Thomas, which was granted in 1944, and it is on an application filed on November 8, 1941.

There has been no contention below and apparently there is no contention here that this patent fails to show this specific material that is being claimed by this application as novel in lubricating oil.

I would like to also call your attention to the patent, the statement on page 2 of the patent, which states in the second, column, just before the claims:

That this application is a continuation in part of our copending application, Serial No. 401,960, filed July 11, 1941.

Under normal circumstances this form means that this patentee is entitled to the date of July 11, 1941, for its disclosed subject matter.

In addition to rejecting all the claims as anticipated by this Cook patent, the Examiner rejected the claims on a combination of three other patents, which appear in the folder: The Salzberg patent at E, the McNab patent at C, and the Wilson patent at F, on the ground that Salzberg discloses the addition to a lubricating oil of an analogous material. It is not the identical material as was

explained here but it is analogous, and the contention is made that it would be not invention to use the particular material that the plaintiff used because of the disclosure in the Salzberg, McNab, and Wilson patents, describing the use of other agents in a lubricating oil composition, such as extreme pressure agents, or pour point depresses, corrosion inhibitors, and things of that sort, which
11 are set forth particularly in some of the more detailed claims.

So I think we can consider this case as depending solely on the use of the zinc, capryl alcohol, and di-thio phosphate material as the novel element of composition.

Now, plaintiffs' counsel has called your attention to the fact that effort was made below to introduce evidence about antedating the Cook, et al., patent, and he has indicated here that efforts will be made to do the same thing.

I would like to object and put him and Your Honor on notice that I will oppose the introduction of evidence about antedating that patent at this time on the ground that he is estopped to do so.

The rule has been stated by the Court of Appeals in the District and other jurisdictions that an applicant who comes to the Patent Office must present all the evidence he has at his command at the earliest possible moment and not wait an unreasonably undue time before he decides to present it.

His deliberate withholding of the evidence, we maintain, has estopped him from presenting it in this proceeding, even though this be a trial de novo.

The Court of Appeals in this jurisdiction has indicated in 4915 actions that they approve such a holding, and I will cite those if you wish them now.

THE COURT: I would prefer them now.

MR. SCHIMMEL: I shall rely for that on the
12 case of Barrett vs. Koppers Company. That is a
case of the Third Circuit, 22 Fed. 2d 395, which

was cited with approval by the Court of Appeals in the District in Schilling, et al., vs. Schwitzer Cummins Company, 79 Appeals D. C. 20, and another case before the court here of Boucher Inventions vs. Sola Electric, 76 Appeals D. C. 160. Certiorari was denied in 383 U. S. 770.

So essentially our position is this: The novelty of the case is anticipated by Cook. Even assuming that Cook does not anticipate, assuming that the Court will permit introduction of evidence antedating Cook, we contend that the specific material is not any different from similar materials used in the prior art, such as Salzberg, and in addition, the answer sets forth in amended paragraph 12 that many of these claims are unpatentable because they do not point out the invention with the particularity that the statute requires, and that objection is particularly applied to expressions, such as "characterizing amounts" appearing in many of the claims. We don't know what that means.

Also the fact that some of the claims set forth a lubricating composition but actually define nothing but this specific thing, capryl alcohol and di-thio phosphate, namely, which cannot be a lubricating composition, but it must be used in conjunction with mineral oil. Many of the claims leave that out.

13 And also with respect to expressions, such as "corrosion inhibitor" which appears in some of the claims, for example, Claim 16, which we contend is so vague and indefinite that we do not know what it means.

Also that the courts have frowned upon defining the composition in terms of their function or characteristics, rather than in terms of what they are. That about sums up our position in the case, Your Honor.

MR. LIMBACH: Your Honor, with regard to the first statement that we are relying entirely in this case for patentability of the claim on the novelty of the zinc salt, I would like to take issue with that because that is not in accordance with our contention.

We are prepared to show by the evidence that there is a decided cooperation between that material and some of the other named materials, materials named in the other claims which will support the patentability of these claims.

With regard to the admissibility of evidence, Mr. Schimmel cited the Koppers case at 22 Fed. 2d 395, and that case has been referred to a number of times wherever this question has come up, and the basis for the statement is that there may not be introduced in the District Court trial under a 4915 action any evidence which was available during the Patent Office proceeding. But that statement is not the law today, and there are a number of
14 cases that distinguish from it. In the case of Minnesota Mining Company vs. Carborundum Company, 69 USPQ 288, where the Circuit Court of Appeals for the Third Circuit held that RS 4915 provides broadly that the trial before the District Court shall be without prejudice to the right of the parties to take further testimony.

I would like to call the attention of the Court to the decision of Judge Letts in the case of Minnesota Mining vs. Coe, 57 USPQ 8, in which Judge Letts held the same way as in the Minnesota Mining against Carbarundum case.

Another case is Clark vs. Resinous Products Company, 75 USPQ 172, which held like the Minnesota Mining case, which it cites with approval, and it also cites a number of additional cases, such as Hoover vs. Coe.

THE COURT: What is the citation of the Clark case?

MR. LIMBACH: Your Honor, that is 75 USPQ 172. It cites Hoover vs. Coe, 69 USPQ 180. That is a decision by the Supreme Court.

Then a very recent decision by the Court of Appeals for the District of Columbia is in Knudsen vs. Gallsworthy, 74 USPQ 324.

Then there are a number of other cases, Jorgenson vs. Ericson, 80 USPQ 261, and Globe-Union, Inc., vs. Chicago

Telephone Supply Company, 103 Fed. 2d 722, and then the case of Minnesota Mining vs. Carborundum Company at 155 Fed. 2d 746.

15 Now, in all these recent cases, Your Honor, it is recognized that the statute specifically provides that the plaintiff may introduce additional evidence in a case of this kind. The Court of Appeals in its decision in the Knudsen vs. Gallsworthy case, in 164 Fed. 2d 509, states this:

"It follows that if the evidence would assist the court, and if no bad faith on the part of the profferer is involved, such as deliberate withholding for some tactical reason, the court could receive the evidence; in fact, as we have already pointed out, the situation might be such as to require the court to seek such evidence."

There has been no deliberate withholding of evidence in the present case. It was offered to the Patent Office for its consideration.

Shall I proceed with the presentation of my case, Your Honor?

THE COURT: Yes.

MR. LIMBACH: In order to facilitate the presentation of this evidence, I have arranged the exhibits in books. They comprise a first volume in which are found all of the originals. In this volume will be found bound and arranged in numerical order the originals of the documents which will be introduced in evidence and from which the witness will testify.

16 Then I have for the convenience of the Court and opposing counsel photostatic copies similarly arranged.

Will you pass that to the Court?

Since it will be noted that there are 42 exhibits in this bound volume, I should like to offer in evidence at this time, as Plaintiff's Exhibit 43, a certified copy of the Asseff application, Serial No. 457,746, which is the application involved in this proceeding.

MR. SCHIMMEL: Is that the complete file?

MR. LIMBACH: That is the complete file.

THE COURT: Is there any objection?

MR. SCHIMMEL: No objection.

THE COURT: It will be admitted as Exhibit 43.

(Thereupon the document was received in evidence as Plaintiffs' Exhibit No. 43.)

MR. LIMBACH: I am ready to go ahead with my witnesses.

THE COURT: I am going to take the verdict from this jury and then we will proceed.

(Thereupon, as indicated, the Court proceeded with other matters, after which the following occurred:)

THE COURT: Can you give me some estimate how long it will take for the presentation of your case?

MR. LIMBACH: We can hardly finish today.

THE COURT: The reason I ask that is because of
the fact the Court meets in general term at half
17 past 3 this afternoon, and it is with considerable
reluctance I adjourn at that time.

MR. LIMBACH: We will be able to finish tomorrow.

THE COURT: It will take most of tomorrow?

MR. LIMBACH: I think so.

THE COURT: Very well. You may proceed.

MR. LIMBACH: I will call as my first witness the applicant Peter A. Asseff, please.

Thereupon

Peter A. Asseff

was called as a witness by the plaintiffs and, being first duly sworn, was examined and testified as follows:

Direct Examination

BY MR. LIMBACH:

Q Are you the Peter A. Asseff, the applicant named in this application, Serial No. 457,746, involved in this proceeding? A I am.

Q Before proceeding with your examination, Dr. Asseff, I should like to have you tell us of your academic qualifications and you academic training. A I have a Ph. D.

THE COURT: Keep your voice up.

THE WITNESS: I have a Doctor of Philosophy Degree in Chemistry from Case Institute of Technology, Cleveland, Ohio.

BY MR. LIMBACH:

18 Q Where are you employed? A The Lubrizol Corporation.

Q Where is it located? A At Wickliffe, Ohio.

Q What is the nature of your position? A I am chief chemist.

Q How long have you been thus employed? A Appromimately 13 years.

MR. LIMBACH: Your Honor, I am going to examine the witness with regard to certain compositions which related to the issues. For your convenience in following the examination of the witness, I have compiled separate sheets. There are in all eight compositions about which the witness will testify, and a separate sheet is devoted to each composition. At the top of each sheet is given a composition of that example, and directly underneath will be found, in parallel columns, reference to the exhibits and by code number, where these compositions are identified. I think it will be of material assistance to the Court.

THE COURT: Reference to the exhibits as indicated?

MR. LIMBACH: Yes, sir.

Before proceeding with the examination, I presume we should have the reporter mark for purpose of identification the exhibits in this folder so we can consider them marked, and as you will note the pages are all marked at the bottom.

19 THE COURT: Is that the same volume I have before me?

MR. LIMBACH: Yes.

THE COURT: I have instructed the clerk to mark this volume as Plaintiffs' Exhibits 1 to 42.

MR. SCHIMMEL: They are not being introduced in evidence but merely for identification?

THE COURT: The Court will consider them marked for identification only and give you opportunity to make the objection which you desire to make in due course.

MR. SCHIMMEL: Thank you.

MR. LIMBACH: I am handing the witness the volume which contains the originals.

BY MR. LIMBACH:

Q Now, the first composition with which we are concerned is Example No. 1, and I would like to ask the witness by having reference to the exhibits which he has in his hand, and the references thereto and the code numbers, first, to establish the composition of the example as being that which is set forth in this sheet, and then to indicate what tests, if any, were conducted on that composition.

I think this procedure will have a lot of individual questions which otherwise would be time consuming. A Code No. 601-49 of Example 1 refers to Exhibit 19.

On Exhibit 19, No. 601-49 is identified as No. 96K, plus .5 percent of 601-19A, plus 1.67 percent of CDS-4.

20 Now, the following right down the line, in Example 1 for the code number 601-19A, reference is made to Exhibit No. 18.

Next, in Exhibit 18, 601-19A is identified "the same as 601-19, made from 601-18A."

Also on Exhibit 18 is a translation of 601-18A, which reads simply, "Same as 601-18."

Next on the code number line in Example 1 is 601-18, which is translated on Exhibit 17.

Q When you say translated on, what do you mean? Is that an accurate statement? A By translating I mean identified. 601-18 on Exhibit 17 is identified as hydrogenated pentaphen, 5 mols, plus 1 mol of P2S5, refluxed in benzine solution 8 hours, washed in water, and dried under vacuum.

For the identification of 601-19 the same Exhibit 17, refers to it as the calcium soap of 601-18, made by double decomposition from sodium soap with calcium chloride.

Now, the translation or identification is complete, with the exception of the material 96K, which is referred to on Exhibit 29. Exhibit 29 identifies Code No. 96 as being crown central Diesel base oil.

Q In the identification of 601-18, Code No. 601-18, you made reference to pentaphen. What is that material, if you know? A That is tertiary amyl phenol.

21 Q And also in your testimony at first given, you referred to CDS-4. What is that material? A That is the calcium salt of diochlor stearic acid.

Q It is the calcium salt of diochlor stearic acid? A Yes, sir.

MR. SCHIMMEL: What was that identification?

MR. LIMBACH: CDS-4.

MR. SCHIMMEL: I just asked.

BY MR. LIMBACH:

Q How do you know this to be so? A From knowledge of the code designation, knowing what the significance of that is.

Q Now, I would like you to go back, and for the information of the Court I would like to have you tell the Court what these records are that are in this book from which you are testifying? A That is a page from the original book of entry on any sample or preparation made in the Lubrizol laboratories.

MR. LIMBACH: The witness is referring to Exhibit 1.

THE WITNESS: Immediately upon the assignment of an oil composition or the completion of a chemical preparation, the material is assigned a code number and this code number is tied in with the particular preparation.

In the first column, and I refer to Exhibit 1, for example, the third member from the top, 482D-11
22 and the description of the material immediately following.

THE COURT: I do not think the Court's volume of exhibits is complete. I do not find Exhibit 1 in this file.

MR. SCHIMMEL: I don't have that either.

THE COURT: I have been unable to find any Exhibit 17, 18, and 19.

MR. LIMBACH: I am sorry.

They are here, Your Honor, the numbering at the bottom of the page.

THE COURT: Those are the numbers at the bottom of the page, and these other numbers have no significance?

MR. LIMBACH: No, sir, none whatsoever. They are the numbers at the bottom of the page.

MR. SCHIMMEL: What was the project number of 1?

MR. LIMBACH: 482D.

THE COURT: I understand it now.

BY MR. LIMBACH:

Q Let us go back again. What is the heading of the top of the sheet marked project number? Will you explain that to the Court? A The material falling into the category of a particular subject matter is assigned a project number, and all preparations and oil blends made within that, and involving that subject matter, is placed in Project 482D.

Q What are the entries in the first column, which
23 is headed, Sample Number? A We start all projects or sample numbers within that particular project with No. 1 as being the first sample prepared in that project, and then subsequent samples take consecutive numbers, for example, the second one made in that project would be 482D-2, and so forth down the line.

Q What is the data which is entered in the second column under the heading, Description? A That is a brief description of the material assigned that sample number.

Q What is the data which is entered in the column headed, Tests? A That is a brief summary of the tests which were scheduled on that particular sample.

Q What is the significance of the entries in the column marked or headed, CK? A CK would indicate that the particular tests had been complete and checked, and marked as being completed. For example, at the top of Exhibit 1, on Sample 482D-6A, it has sol, sol for soluble, and the test was started on 9-6-39.

The sample itself was prepared on 9-6-39, and the tests started on 9-6-39, and completed on 9-7-39.

Upon the completion of the test a check mark is put in the check column.

24 Q I believe that that type of recording is uniform down through Exhibit 28; is that correct? A Yes.

Q Now, will you please refer to Exhibit No. 29 and tell the Court what that is? A Exhibit 29 gives the identification of certain code numbers which were chosen to identify a particular product because of the lengthiness of the name involved. We designate a particular thing by a code number.

At the top of Exhibit 29, for example, Code No. 80 is an abbreviated name, you might call it, for Barnsdall Be Square SAE 30, and so on and down the line, and those are code numbers for base oils.

Q How were they kept? A There is a place where a person's name has been signed, and it is notarized upon the assignment of a code number to a particular material.

As on Exhibit 29, the number is attested to before a notary public as being the code number for the described particular material.

Q Now, how are these records kept in the Lubrizol Corporation? Are you familiar with that? A With the records on Exhibit 29?

Q Yes. A These records are kept in the office of the Lubrizol Corporation.

25 Q Under whose jurisdiction are they kept? A These have been under the jurisdiction of Mr. Kelvin Smith.

Q Is he in the courtroom? A Yes, sir.

Q Will you refer to Exhibit 30? Is that type of record similar to 29? A Yes, sir.

Q Is the same true as to 31, 32, and 33? A Correct. These are all code numbers with subsequent translations of what the code number means.

Q Now, will you refer to Exhibit 34, please? What is this type of record, without identifying the substance? A Exhibit 34 is a summary of the results of engine tests on particular combinations.

Q What type of engine was used in running the tests reported in Exhibit 34? A Lauson engines; Diesel test procedure.

Q I hand you a copy of your application involved in this proceeding, on page 8 thereof, this being Plaintiffs' Exhibit No. 43, and call your attention to a description of an engine test.

Will you review that, please? Is that procedure or test procedure there described similar to the ones followed, for example, in arriving at the data reported in

26 Exhibit 34? A Yes, it is.

Q Now, will you refer to Exhibit 35 and tell the Court what that is? A Exhibit 35 is a summary of engine tests, results of Lauson Diesel engine tests, results on particular blends of material and lubricating oil.

Q Were those tests the same type as those outlined in the application to which you have just referred? A Yes, they are.

Q How about Exhibit No. 37? We will skip 36 for the time being.

I might say that the original of Exhibit 36 is in the book which the witness has. It was inadvertently overlooked in the preparation of the photostatic copies for the other books. We will try to get those by tomorrow morning, Your Honor.

Will you now refer to Exhibit 37? A Yes, sir.

Q And any other similar sheets which you can identify as giving the same type test data as the engine tests reported in Exhibits 34 and 35 which you have just identified. A Exhibit No. 37 gives the same type data.

Also Exhibit 38, Exhibit 40, Exhibit 41, and Exhibit 42.

Q I notice that the printed form on which these data
are reported varies from exhibit to exhibit. Can
27 you explain the reason for that variation? A Re-
ferring to the Lauson Diesel?

Q That is right, the exhibit you just referred to. There seems to be a difference in the printed form used. A Yes.

A comparison of Exhibit 38 with Exhibit 42, for example, Exhibit 38 does not provide for a continuous record of each 30-hour inspection of the Lauson Diesel results, whereas Exhibit 42 makes the provision every 30 hours for recording the result of the inspection.

Q It is merely, therefore, a difference in the setup for getting the data? A That is correct.

Q While we are on the Lauson Diesel sheets, and I have referred to them in that way for convenience, what is the significance of the various entries there?

Let us go back, for example, to Exhibit 34, so the Court may understand the nature of the tests, the results of which are given there. A The first example under the general identification, Texaco SAE 30, OR-3084, which is in the first column, and containing as indicated in the second column, 1 percent of compound No. 7 and 1 percent Paranox.

Underneath that, 60 designates the duration of the test
in hours, and the inspection of the engine at that
28 particular time during the course of the test.
Immediately underneath No. 60, piston rating,

B-plus, A being the highest rating assigned, B second, and B-plus would signify that it is between A and B somewhere, higher than B, closer to the A.

Oil filling, zero.

Q At this point I would like to interrupt to show the Court the originals, the photographed charts, which in reproduction in black and white in Exhibit 43, lost their color.

This is another copy of a chart.

MR. SCHIMMEL: That is the same as the one in the record?

MR. LIMBACH: That is right. Upon referring to the colored photographs, A, B, C, D, and so forth, there is an indication of the condition.

BY MR. LIMBACH:

Q Will you proceed, please? A Percent of oil filling means the extent to which the oil ring is filled with sludge.

Rings stuck means number of compression rings stuck in their grooves.

The bearing weight loss in milligrams, and is the total weight loss of the bearing, tying the piston to the crankshaft of the engine. It has the bearing weight prior to the beginning of the test and at intervals, at the different hours indicated.

29 It shows a weight loss of 328 milligrams in the particular bearing, and you can see in 120 hours the corresponding rating for the same test and in the same engine, that the piston went from B-plus to E-minus, I believe it is.

And the oil filling measures zero; ring stuck, zero, and the bearing weight loss went up to 814 milligrams.

Below the engine condition section is the drain analysis. At the conclusion of the tests, the oil is drained from the crank case of the engine and subjected to the analysis described in the drain analyses section, after which we have the results listed in the first column for the particular test, for the example described at the top of the first column.

Percent of viscosity increase, 114, which signifies that the oil increased in viscosity 114 percent over that of the original oil. It means it thickened up to a measurable extent, 114 percent of the original oil.

Conradson carbon residue, 3.78, as compared to the original base carbon residue of 49.

Naptha insolubles, 51.7.

Q Are these tests that you have just referred to as Conradson carbon, are they well known tests in the lubricating oil industry? A Yes, sir.

Q Will you proceed? A Another test described
30 in the drain analysis for which we have a result is
the acid number,. or quite frequently referred to as the neutralization number of the lubricant. It is a measure of the acid content upon the acid side, and the measure of the basic side, if it happens to be on the basic side of the indicator.

Q Will you refer back to Plaintiffs' Exhibit 18? There I notice it refers to Code No. 601-19A and 601-18A, and then back on Exhibit 17 we find entered Code No. 601-18 and 601-19.

What is the relationship between the two code numbers like these pairs just referred to, which differ from each other only by a letter which follows? A According to the system which we use, the first number, the first test made of a particular type would get a number, 601-18, and if that preparation is repeated again after the first time, the repeat would be assigned No. 601-18A, which means that is the second preparation of that particular type of material.

Q What about the material itself? Are the particular materials prepared in the same way? A Yes.

Q So they would as nearly as possible be identical materials; is that right? A That is correct.

Q Now, we have a general review of the exhibits
31 behind us. I would like to have you proceed then
with a further discussion of Example 1, which was

started a moment ago, and indicate how you determine from the records of Example 1 its average composition given on the sheet you have before you.

A On Example 1, under the code number first referred to, 601-49, I have in the right-hand column, Exhibit No. 19.

We turn to Exhibit No. 19, and I see that Code No. 601-49 is described as No. 96K, plus .5 percent of 601-19A, plus 1.67 percent of CDS-4.

Now, the problem comes in identifying what the various code numbers are which describe the sample.

So that the next exhibit referred to along this line of identification is Exhibit 18, which says that 601-19A is the same as 601-19, made from 601-18A.

And 601-18A on Exhibit 18 is described as being the same as 601-18.

Now, over to Exhibit 17 for a description of 601-18 and 19. 601-18 is a product prepared by the reaction of 5 mols of hydrogenated pentaphen, plus 1 mol of P285, refluxed in benzene solution for 8 hours, washed with water and dried under vacuum.

601-19 in the same exhibit is identified as calcium soap of 601-18, made by double decomposition from sodium soap with calcium chloride.

Q What is meant by the double decomposition? A
First reacting the acid with sodium hydroxide, in
32 this case to form the sodium soap or salt, and subsequent reaction with the solution of calcium chloride, causing a precipitation of calcium soap which is insoluble in water, sodium salt being more soluble than calcium salt.

Q What is precipitated? A The calcium soap of that particular material.

Q Is that a well known method for preparing soaps of this kind? A Yes, sir.

Q Will you proceed? A In Exhibit 19, under 601-49 reference is made to No. 96K, as being one of the constituents of that particular composition, and No. 96K is described in Exhibit No. 29. Exhibit 29 shows the Code No. 96.

Now, to give you an explanation and the reason for the letter K. Each subsequent batch of No. 96 material would be assigned a subsequent letter. The code number 96 is the first material received, 96A would be the second of the same type, and so forth, down to the letter K in this case, and that is described as Crown Central Diesel base oil.

Q How do you establish the composition percentagewise as it is given at the top of the page? A We have established from 601-49, Exhibit 19, that—

Q (Interposing) Will you please refer to the
33 Lauson Diesel sheet, Exhibit 37? A All right.

Q What does that show? A It shows that 601-49, which is the code number of this material in question—first of all is a blend in Crown Central 30 of the calcium salt of the amyl cyclohexanol derivative, at .50 per cent concentration plus .50 percent CDS.

Q What does that mean, calcium salt? A It means the calcium salt of the reaction product of P2S5 and hydrogenated pentaphen, and pentaphen, being amyl phenol, it then is the calcium salt of the reaction product of P2S5, and amyl cyclohexanol.

Q That is what is meant by the entries calcium salt? A That is correct.

THE COURT: I am going to have to adjourn at this time because of a general term meeting.

Mr. Clerk, we will meet in our own courtroom tomorrow morning?

THE DEPUTY CLERK OF THE COURT: That is correct, Your Honor.

THE COURT: Our hearing tomorrow morning will be in the courtroom at the other end of the corridor.

(Thereupon, at 3:30 o'clock p.m., a recess was taken until 10 o'clock a.m., on Wednesday, May 18, 1949.)

34 UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF COLUMBIA

PETER A ASSEFF, et al.,
Plaintiffs,
vs.
LAWRENCE C. KINGSLAND,
Commissioner of Patents,
Defendant.

Civil Action
No. 3967-47.

Washington, D. C.,
Wednesday, May 18, 1949.

Pursuant to adjournment theretofore taken, the trial of the above-entitled case was resumed at 10 o'clock a.m., on Wednesday, May 18, 1949, in the United States District Court for the District of Columbia, in the Courthouse, at Washington, D. C.

BEFORE:

HONORABLE EDWARD A. TAMM, District Judge of the United States District Court for the District of Columbia.

APPEARANCES:

ALMON S. NELSON, ESQUIRE, and OSCAR C. LIMBACH, ESQUIRE, on behalf of the plaintiffs; and

JOSEPH SCHIMMEL, ESQUIRE, on behalf of the defendant.

35 *Proceedings*

THE DEPUTY CLERK OF THE COURT: Asseff, et al., vs. Kingsland.

THE COURT: You may proceed.

MR. LIMBACH: Dr. Asseff, take the stand.

Thereupon,

Peter A. Asseff

resumed the witness stand pursuant to the adjournment and testified further as follows:

Direct Examination (Resumed)

BY MR. LIMBACH:

Q Now, at the conclusion of yesterday's session, Dr. Asseff, you were referring to Example No. 1, and in your testimony with regard to its composition reference was made to a material which you mentioned as CDS.

I hand you a sheet of paper which I shall ask the reporter to mark for identification as Plaintiffs' Exhibit No. 44.

(Thereupon a document was marked Plaintiffs' Exhibit No. 44 for identification.)

BY MR. LIMBACH:

Q I ask you what that is. A This is the code sheet identifying CDS as calcium dichlor stearate. The sheet is dated 9-30-27.

36 Q When you say the code sheet, is that part of the records of the Lubrizol Corporation? A Yes, it is.

Q Are you familiar with those records? A Yes, I am.

MR. LIMBACH: I offer in evidence this Exhibit No. 44. I am sorry I do not have copies of it. Will that go in all together?

MR. SCHIMMEL: I object to that.

BY MR. LIMBACH:

Q In your testimony with regard to the same composition, you also referred to a material by the name of Pentaphen.

I hand you a catalogue and ask you if you can tell me what it is?

Let us have it marked for the purpose of identification as Exhibit No. 45.

(Thereupon a document was marked Plaintiffs' Exhibit No. 45 for identification.)

THE WITNESS: This is a catalogue put out by Sharples Chemical Company, and the date of the catalogue is December, 1939.

On page 19 the material described as Pentaphen is identified as para tertiary amyl phenol.

BY MR. LIMBACH:

Q Now, to go forward with the more specific identification of the composition of Example 1, what was
37 the precise composition of that example as shown by the code numbers about which you testified yesterday? A Under Code No. 601-49, the identification is No. 96K, which we identified by a series of code numbers as being Crown Central base oil, plus .5 per cent of 601-19A, which we have identified through the series of code references as the calcium salt of P2S5, amyl cyclohexanol or hydrogenated pentaphen, plus 1.67 per cent of CDS-4, which we have identified as calcium dichlor stearate oil solution, 1.67 per cent being equivalent to .50 per cent of the pure salts, since this material CDS-4 is a 30 per cent solution in oil.

Q What was the composition in Example 1—that is, I would like to have you tell the Court the complete composition, how much mineral oil and how much of the various chemicals. A The composition was mineral oil 99 per cent, calcium dichlor stearate .5 per cent, calcium salt P2S5, treated amyl cyclohexanol .5 per cent.

Q Will you please refer to Exhibit 19 on which appears Code No. 601-49, and will you please tell the Court how those entries were prepared and the significance of the dates which appear in the last three columns? A Sample Code 601-49, according to the code system which I explained yesterday, was made up on 4-1-40, and the Lauson Diesel test scheduled.

38 The sample was sent to the engine laboratory on that date, 4-1-40, and the test started as indicated on 4-1-40, which is the date that sample was sent to the engine laboratory.

In the completed column at the extreme right-hand portion of Exhibit 19, under 601-49 is the date, 4-9-40. This date signifies the date upon which the test, the Lauson

Diesel test was completed as indicated by the engineering laboratory.

Q In whose handwriting are the entries on Exhibit 19? A In my handwriting.

Q Will you explain the mechanics of making entries as to dates? In other words, what sequence was followed in entering those dates in this exhibit? A Under the column made, the date was entered on the date this blend was completed and put together.

Under the starting date, the date upon which the completed blend was sent over for testing to the mechanical engineering laboratory.

Under the completed column appears the date which indicates the time at which the test which was scheduled was completed.

So that we have a date made for the sample, a date started for the test, and a date completed for the test
scheduled in the column adjacent to the Lauson
39 Diesel, which indicates which test was scheduled or
started and the date completed.

Q I should like to have you run through Exhibits 1 to 28 and tell the Court whether what you have just stated with regard to Exhibit 19, as being prepared by you in your own handwriting and in accordance with the procedure just outlined, applies to all of the other of those Exhibits 1 to 28? A It applies to 1, 2, 3, to 4, to 5, to 6, to 7, and the majority of 8 with the exception of two entries at the top of this exhibit, identified under Sample No. 526-29D7 and 526-29D8.

Q Do you know who made those entries? A I recognize those entries as being made in the handwriting of Mr. Frey.

Q Will you go ahead with the rest of them? A All of Exhibit 9, all of Exhibit 10;

All of 11, all of 12, all of 13, all of 14, all of 15, all of 16, all of 17, all of 18, all of 19, all of 20;

All of 21, all of 22, and part of 23.

Those made in other than my handwriting are described as Sample No. 601-302 and 601-304.

Q Who made those? A I recognize those as the handwriting of Dr. Drake, W. B. Drake.

40 Q What was the occasion for his making those particular entries? A He was in charge of the preparation of those compounds at that time.

Q Were you familiar with the work when it was done? A Yes, these code records were kept in my office and kept in there to make these entries.

Q Could you see the entries at or about the time that they were made? A Yes, sir.

Q Will you proceed? A All of 24, with the exception of 601-302A, which is the fourth sample from the top of the page. That entry likewise was made by Dr. Drake.

Q Were you familiar with it at the time it was made? A Yes, sir.

On Exhibit 25 there are two entries which are in my handwriting, 601-313, which is the third entry from the top, and 601-303D1, which is the fourth entry from the top, and the rest of the entries on this exhibit were made by Dr. Drake.

Q And are you familiar with those at the time they were made? A Yes, sir.

On Exhibit 26, two entries are in my handwrit-
41 ing, and the rest being Dr. Drake's.

The two in my handwriting are 601-301D1, which is the second sample from the top, and 601-317, which is the third from the top.

Q Do you know who made the other entries? A Yes, Dr. Drake made those.

Q Are you familiar with that and the work at the time? A Yes, sir.

Q Go ahead, please. A On Exhibit 27, the entries in my handwriting are 601-313D1, which the second entry from the top of the page, and 601-321, which is the fourth entry, and 601-321A, the fifth entry, and 601-322, the sixth entry.

The other two were made by Dr. Drake, again.

Q And were you familiar with that work at about that time that is represented there? A Yes, sir.

On Exhibit 28, the entries in my handwriting are 601-353, the second entry from the top, and 601-353A, the third entry from the top;

601-343D1, the fourth entry from the top, and 601-342D1, following 343, and 601-355, at the bottom of the page.

The other three entries are in the handwriting of Dr. Drake.

42 Q And were you familiar with this work at the time? A Yes, sir.

Q At the time those entries were made? A Yes, sir.

Q Now, will you refer, please, to Exhibit 37, which you stated in your testimony yesterday was the record of the engine test run on Exhibit No. 1.

Will you identify which composition on Exhibit 37 is the Sample No. 1? A The extreme left-hand column on Exhibit 37 carries a series of code numbers, one describing the composition on Exhibit 1 as 601-49.

Q That is the sixth from the top? A Correct.

Q Will you please indicate what is the significance of the entries following that exhibit? A In the column under identification is a brief description of the composition represented by 601-49 and as given it says that it is a half per cent of calcium salt of the amyl cyclohexanol derivative, plus a half per cent of CDS.

Q Is there any oil present in that? A All samples are in Crown Central 30, as indicated at the top of the identification column.

Q What is the significance of the other entries?

43 A The other entries are a summary of the engine test itself, a record of the condition of the engine at various hours throughout the test, and this shows at 30 hours, the piston condition, C;

Ring stuck, 1 and 2;

Oil ring filling, sludge 50 per cent;

And bearing, under B, 314 miligrams loss of weight.

Q Well, now, take up Exhibit No. 2 as you did yesterday with regard to Exhibit No. 1, and in order to save the time of the Court, without further questions on my part, will you go through the designated code numbers identified on the samples?

MR. SCHIMMEL: May I suggest, the time of the Court may be saved if we eliminate the detailed explanation until you get to the exhibit with which you are actually claiming it is related to the claims. I don't think this particular matter is material to the issue involved here. I think we are wasting time.

MR. LIMBACH: I do want to get these examples in, Mr. Schimmel.

Are you willing to stipulate that Exhibits 1, 2, 3, 4 and 5 have the composition shown on these white sheets and were tested at about these dates?

MR. SCHIMMEL: No.

MR. LIMBACH: If not, I will have to go ahead
44 with the examination.

THE COURT: Are you willing to make that stipulation?

MR. SCHIMMEL: I cannot stipulate to that.

THE COURT: You may proceed.

BY MR. LIMBACH:

Q Go ahead, please. A According to Exhibit No. 19, which identifies Code No. 601-50, 601-50 is indicated to be No. 96K, plus a half per cent of 601-19A, plus 1.67 per cent of 482D-18P.

Now, the first two parts of that description, 96K and 601-19A, we have already identified.

There remains to identify 482D-18P, and referring back to Exhibit No. 5, we will find that 482D-18P is the same as 482D-18N and running right down the line is the series of letters attached to the Code No. 482-18, which will work us all the way back to 482-18 itself, which is in Exhibit 1.

Under 482-18 on Exhibit 1, the second sample from the bottom, the material described is cobalt, CPSA, made as a 30 per cent concentrate in No. 38 oil, by double decomposition, and in parenthesis indicated as the cobaltous salt of CPSA.

Now, to the de-coding sheet, Exhibit 30, to identify the letters CPSA, and in the second section from the bottom,
the second code from the top of that section are
45 the letters CPSA, and under description this material is identified to be chlor phenyl staaric acid.

I have described the parts as sections, the sections having a notary seal at the bottom, and the second section from the bottom is the portion in which the CPSA appears.

Now, having established what the code CPSA means, sample 482-19 is the cobalt salt of chlor phenyl stearic acid, 30 per cent concentration in 38 oil.

So back to Exhibit 19, under 601-50, the composition is base oil, Crown Central oil, plus a half per cent of calcium salt of P2S5, treated cyclohexanol, plus a half per cent of the cobaltous salt of chlor phenyl stearic acid.

Q Were engine tests run on that composition? A Yes.

Under Lauson Diesel sheet, Exhibit 37, and under Code No. 601-50 are the results of the engine inspection at 120 hours. That would be the seventh sample from the top.

Q When was that engine test run and how do you establish that date? A The date for the run on the engine test is established on Exhibit No. 19.

According to the system which we have previously explained, the sample was made on 4-1-40, and the date the test was completed was 4-12-40.

Q 40 is significant of the year 1940? A That
46 is correct.

Q Now, going on to Example No. 3, please. A On Exhibit 21, for Code No. 601-100, under the column headed Description, you have that sample identified as No. 96K, plus 1 per cent of 601-98.

Now, we have already established the identity of 96K, and it remains to establish the identity of 601-98.

Referring to Exhibit 21 again, it shows this sample coded at 601-98 to be a zinc salt of 601-96, and 601-96 appears at the top of Exhibit 21 and is identified as the reaction product of octyl alcohol, 5 mol, plus P2S5, 1 mol, refluxed in benzine solution, at 100 degrees Centigrade for 4 hours, washed with water, and dried in vacuum.

That identifies 601-98 as the zinc salt of the reaction product of octyl alcohol and P2S5.

Q What was the composition of that material? A 99 per cent mineral oil and 1 per cent of the zinc salt of the reaction product of 5 mols of octyl alcohol and one mol of P2S5.

Q Was an engine test run on that? A On Lauson Diesel sheet, Exhibit 38, under 601-100, the result of the test at 30 hours: B, zero, zero, 12.

These codes designate the same thing that I explained previously in describing the engine condition.

At 90 hours it is: C, zero, 25, and 36.6.

Q When was the engine test run? What item
47 would it be? A The date is established on Exhibit 21, and according to the system previously described, for sample 601-100, the composition was prepared on 4-30-40, and the test completed on 5-20-40.

Q Will you proceed with Example 4? A Referring to Exhibit 7 for Code No. 526-17, one finds that that composition is described as 526-10, plus 1 per cent of 564C-33F, plus .5 per cent of 601-91F.

Now, 526-10 is described on Exhibit No. 6 as being Texaco uninsulated SAE 30.

Q What is that? What is Texaco uninsulated SAE 30? A That is a SAE 30 grade base oil, containing no additional treatment, other than just the mineral oil base.

Q Go ahead. A 564C-33F is described on Exhibit 14 as being the same as 564C-33, and in order to describe that we have to go back to Exhibit No. 13.

On Exhibit 13, at the top of the page, Code No. 564C-33 is described as magnesium salt of 564C-23, made by the reaction of magnesium in methyl alcohol solution; theoretical amount employed; refluxed for five hours at 80 degrees Centigrade.

In order to identify 564C-23, we go back to Exhibit 12, and under Code 564-23, the description refers to 1
48 mol of salicylic acid, plus 1.15 mol of lorol alcohol, heated in xylene solution at 150 to 160 degrees Centigrade for 18 hours. In addition, present during the reaction is 2 per cent of fuming sulphuric acid, added over an 18-hour period.

At the end of this time the material was washed with water, the xylene distilled off, and the residue extracted with dilute sodium hydroxide, NAOH. The portion insoluble in NAOH taken as product. Material dried using benzine.

Now, 564C-23 is described as the lorol ester of salicylic acid and going to 601-91F on Exhibit 22, we find that to be the same as 601-91, which is described on Exhibit 20.

601-91 on Exhibit 20 is described as zinc salt of 601-90, prepared like, made like 601-65.

And 601-90 on Exhibit 20 is described as the reaction product of methyl cyclohexanol, 5 mol, plus P2S5, 1 mol, made like 601-18, which we have already described.

That completely identifies composition 526-17, and we refer back to Exhibit 7, that translates 526-10 to mean Texaco uninsulated 30, plus 1 per cent of 564C-33F, which we have identified as the magnesium phenate of lauryl salicylate, plus a half per cent of 601-91F, which we have identified as the zinc salt of P2S5, methyl cyclohexanol.

Q Were engine tests run on that? A Reference to the engine test is found on Exhibit 34, and the third
49 column to the right, from left to right, shows sample 526-17.

At the top of the column it is identified as 1 per cent of compound number 7, and a half per cent of compound number 6, with the engine test described below.

At 60 hours the piston rating, oil filling, number of rings stuck, weight loss in milligrams is given, and at 120 hours the same data is given.

There is a subsequent drain analysis of the oil sample taken from the crankcase of the engine at the end of the test.

Q When was that test run? A That date is obtained from Exhibit No. 7.

Under 526-17, it shows that the composition was prepared on 8-1-40, and the test completed on 8-9-40.

Q Go ahead then to Example No. 5, please.

BY THE COURT:

Q This figure on Exhibit 7, relating to Sample 526-17, is that 8-1-40 under column "made" or is that a "5"? A That is an "8", sir.

THE COURT: Very well.

BY MR. LIMBACH:

Q Go ahead, please. A Example 5, under Code No. 526-55, by referring to Exhibit No. 9, 526-55 is identified
as 526-11A, plus 10 per cent of C-93 by volume, and
50 in identifying 526-11A, we refer to Exhibit 8, that
describes 526-11A as Texaco uninsulated SAE 20.

For identification of C-93, referring to Exhibit No. 33, the second code from the bottom, identifies C-93 as Lubrizole concentrate No. 737, which is 8 parts ossa 20 by volume, contains 3.2 per cent sulphate ash, and 2 parts of 728 by volume, the composition to be used at 10 per cent by volume.

Now, in identifying Lubrizol No. 728, referred to as C-87, which is on Exhibit 33, the second code from the top of the page, which identifies Lubrizol concentrate 728 as 46.0 per cent ZMP; 54.0 per cent Pa. 150 neutral by weight.

For a description of what is meant by the code ZMP, refer to Exhibit 31, and in the section at the bottom of the page, the second code from the top of that section, ZMP is zinc salt of methyl cyclohexanol, P2S5 reaction product.

BY THE COURT:

Q You say in the document, which is Example 5, that C-87 is identified in Exhibit 32, and your testimony refers to Exhibit 33. Are both statements correct? A C-93 is identified on Exhibit No. 33.

Q I am asking about C-87. You refer to Exhibit 32 and identify your testimony from Exhibit 33. A C-87 is identified on Exhibit No. 32.

MR. LIMBACH: It is on both exhibits, Your Honor.

THE COURT: Very well.

51 BY MR. LIMBACH:

Q I think you had gotten as far as C-90. A ZMP, I have finished that also, on Exhibit 31, describing that as the zinc salt of methyl cyclohexanol, P2S5 reaction product.

Q In your identification of the one component of this composition, you referred to the composition as ossa 20. I hand you a sheet which I would like to have marked for identification as Plaintiffs' Exhibit 46.

(Thereupon the document was marked Plaintiffs' Exhibit No. 46 for identification.)

BY MR. LIMBACH:

Q I ask you what that is. A That is a part of a report on this particular subject matter.

Q Is it a company report from company files? A Yes, sir; it is a company report from company files.

Q What was this, an aid in identification of the material? A Ossa 20 is described on the report as calcium salt of the sulphonic acid.

Q Is there any indication of the derivation of the sulphonic acid? A That is obtained when preparing mineral white oil.

Q Would that be a petroleum sulphonic acid?
52 A Yes, sir.

Q Would it be calcium salt of petroleum sulphonic acid? A Yes, sir.

Q Was an engine test run on that composition? A On Exhibit 35, Code No. 526-55, which is the last test on that sheet, the results of the engine test are given, together with the drain analysis on the sample at the end of the test.

Q When was that test run? A Referring back to Exhibit 9 for the date, 526-55, the composition was prepared 6-23-41. That is at the top of the page.

And the test, the Lauson Diesel test, was completed 7-21-41.

Q I don't believe you told us yet the precise composition percentagewise of that. A Referring to Exhibit 9, that is Texaco SAE 20 base oil, plus 10 per cent of C-93 by volume, and I have gone through identifying C-93 on Exhibit 33, which describes this C-93 as being Lubrizol concentrate 737, which is 8 parts of ossa 20 by volume, and 2 parts of 728 by volume.

We have identified 728 as C-87 on Exhibit 32, which means this composition C-93 when used at 10 per cent by volume will give you 8 parts of ossa 20 by volume
53 and 2 parts of 728 by volume, and on the basis of the pure chemical would yield 1.6 per cent of calcium petroleum sulphonate, with 1.0 per cent of zinc salt of P2S5 treated methyl cyclohexanol.

Q In how much oil? A 97.4 per cent of mineral oil.

Q Will you now proceed to Example 6, please? A Referring to the Code No. 601-313, on Exhibit 25, I have the material under Code No. 601-313 identified as No. 106 oil plus 1 per cent of 601-302, plus .75 per cent of 544-107.

Now, in identifying that 601-302, turning to Exhibit 23, you will find that identified as the reaction product of capryl alcohol and P2S5, 5 to 1 mol, refluxed at 100 degrees Centigrade for 8 hours, followed by the addition of an equivalent amount of zinc oxide added at 90 to 100 degrees Centigrade, stirred for one hour at 150 to 160 degrees, and dissolved in benzine, filtered, and the benzine removed under vacuum.

And for a description of 544-107, turning to Exhibit No. 11, which is the second sample from the top of the page, it shows 544-107 being designated as a number 7 soap, with a code number, and turning now to Exhibit 36—

Q (Interposing) At that point, I would like to interrupt.

Exhibit 36 is the one of which no photostat was 54 available at this time, and it was not possible to have them made last night.

THE COURT: What is the nature of the exhibit? Is it an engine test?

MR. LIMBACH: It is an identification of the No. 7 soap as being a specific chemical.

BY MR. LIMBACH:

Q Go ahead. A On Exhibit 36, 564C-292 is described as No. 7 soap, made from 564C-286, and on a preparation report for 564C-292, a description is provided for the preparation of magnesium lauryl salicylate, using magnesium oxide .

THE COURT: Has counsel seen the exhibit?

MR. SCHIMMEL: I have not.

THE COURT: Do you want to examine it; if it will make the testimony any more clear to you?

MR. SCHIMMEL: I think I can follow it.

BY MR. LIMBACH:

Q Proceed, please. A We identify No. 7 as the magnesium salt of lauryl salicylate.

That then indicates the composition of 601-13 to be 98.25 parts mineral oil, and .75 per cent of magnesium phenate of lauryl salicylate, and 1 per cent of zinc salt of P2S5 treated capryl alcohol.

55 Q Was an engine test run on that composition? A Yes.

Referring to Lauson Diesel sheet, Exhibit 40, one finds under Code 601-313 the test results on the Lauson engine for this particular sample. It would be the fourth sample from the top of the page.

Q Have you any special comment with regard to the data which follows on that Exhibit 40? A It shows this test to be quite satisfactory.

Q The example which follows—refer to Exhibit 40 again, please. A Yes, sir.

Q The example on Exhibit 40 which immediately follows 313, namely, Example 321, is that similar to 313? Was that similar to 313? A Yes, with the exception that the zinc salt of the alcohol P2S5 product was derived using normal octyl alcohol in 321, rather than capryl alcohol as in the case of 313.

Q So the two compositions are identical with the exception of the alcohol used in preparing the salt? A That is correct.

Q In 313 that alcohol was what? A Capryl alcohol.

Q In 321 the alcohol was what? A Normal octyl alcohol.

56 Q Will you go ahead with Example No. 7, please? A Example 7 shows the composition described under Code No. 601-310.

On Exhibit No. 24, under 601-310, which is the third sample from the bottom of the page, 601-310 is identified as 737 concentrate prepared from 601-302A; 80 per cent C-90B, plus 20 per cent of 601-309 by volume.

Now, on Exhibit 32, C-90B (B being an indication that this is a repeat batch of C-90, as was previously explained), and that is the second code number from the bottom, and it shows C-90, on Exhibit 32, to be ossa 20.

Q Is that the same ossa material you identified before as calcium petroleum sulphonate? A Yes, sir; 20 per cent solution in oil.

Referring to the Code No. 601-309 on Exhibit 24, it shows that to be 728 concentrate prepared from 601-302A, and the composition given as 46 per cent by weight of 601-302A, plus 54 per cent by weight of number 7C oil.

In identifying 601-302A, Exhibit 24 shows that to be a repeat batch of 601-302, that is the fourth sample from the top of the page on Exhibit 24.

Referring to Exhibit 23 under the description of 601-302, we find that is the reaction product of capryl alcohol and P2S5, 5 to 1 mol ratio, to form acid, neutralized
57 with zinc oxide to form the zinc salt of that reaction product.

So that in substance 601-310 is a composition containing mineral oil 74 per cent, calcium petroleum sulphonate 16 per cent, and zinc salt of P2S5 treated capryl alcohol 10 per cent.

Q Are you familiar with the materials referred to as ossa 20 and number 728 concentrate? Are they materials often used by the Lubrizol Corporation in the preparation of its products which it sells? A Yes, sir.

Q In that connection have you had occasion to determine the specific gravity of these materials? A Yes, sir.

Q Do you know what it is? A The specific gravity of ossa 20 is approximately .89.

Q What is the specific gravity of 728? A Approximately .97.

Q Have you completed the report of the engine test on Example No. 7? A There is no engine test on Example No. 7.

Q What was done with Example No. 7? A That composition was prepared and observed for short periods of storage to determine whether the materials
58 were compatible.

Q What was found as the result of that test? A The materials were found to be compatible at the end of the storage period, beginning on 9-18-41 and ending on 10-2-41, as indicated on Exhibit 24 under the Code No. 601-310. The test scheduled, storage, and the sample was made up 9-18-41; the test started 9-18-41 and was completed on 10-2-41.

MR. LIMBACH: May I have Plaintiffs' Exhibit No. 43, please?

BY MR. LIMBACH:

Q I refer you to Exhibit 43, which is a certified copy of your patent application involved in this proceeding, and specifically to page 8A, whereon is contained Table A giving seven examples of lubricants.

Do you identify any of that table as being similar to Example 7? A Example No. 6 would be an oil blend of the concentrate at 10 per cent by volume, the concentrate described in Example 7, Code No. 601-310 on Exhibit 24.

Q Do I understand you to say that that oil, No. 6, on page 8A of your patent application was prepared by using a 10 per cent oil solution of Example 7 about which you have testified? A Yes, sir.

59 MR. LIMBACH: And the engine tests on No. 7 are therefore already reported in the patent application and we are not putting in those additional records.

BY MR. LIMBACH:

Q Now, go on to Example No. 8, please. A Referring to Code No. 601-321 on Exhibit 27, we find that material is described as No. 106 oil, plus .75 per cent of 544-107, plus 1 per cent of 601-318.

Now, we have already established the identify of 544-107, a previous sample, and there remains to identify 601-318.

Turning to Exhibit 26, the second sample from the botom of the page, and 601-318 is the zinc salt of the derivative of P2S5, plus duPont normal octyl alcohol, made by the regular procedure for No. 6.

Q When you say No. 6, what do you mean? A Zinc salt of the reaction product of P2S5 alcohol, 5 mol of alcohol to 1 mol of P2S5.

Q Is No. 6 the code used in identification of that material? A Yes.

Q Go ahead. A We identify sample 601-321 on Exhibit No. 27 as 98.25 per cent mineral oil; .75 per cent

magnesium phenate of lauryl salicylate; and 1 per
60 cent of zinc salt of P2S5 treated octyl alcohol, nor-
mal octyl alcohol.

Q And were there Diesel engine tests run on this? A Yes.

Referring to Lauson Diesel sheet, Exhibit 40, we find under the last two samples on the page, 601-321 and 601-321A. The second sample signifies a repeat test of that composition, and the results of those two engine tests are given.

Q When was that engine test run? A 601-321, referring to Exhibit 27, shows that composition to have been prepared on 10-8-41, and the test completed on 11-3-41.

Q Now, not only with regard to this particular pair of exhibits, but also referring to the patent and the engine test on Exhibit No. 8, and also reference to the pair of exhibits on the other examples, I notice the date entered in the code sheet on Exhibit No. 27 is different from the date which is typed in on the Lauson Diesel sheet. How do you account for that difference? A Referring to Exhibit 40, the date at the top of the page is the date upon which the test pertaining to this particular subject matter were summarized and written up in the form of a report, which is Exhibit 40. That is the date of the report.

Q And then how do you fix the date of the com-
61 pletion of the test? A The date of the test, re-
ferring back to Exhibit 27, from our records, it
indicates the date upon which the particular composition was prepared as indicated under the column—the last three columns on the right.

The first one in the group indicates the date on which the composition was made. The second indicates the date upon which the test was started, or assigned, and the last column indicates the date upon which the test was completed. This is a continuous record of all tests scheduled.

Q And it is made at what time with relation to when the work was actually done? A It is an accurate record of time at which the tests were done.

Q Was the date entered at the time that the work was done, or weeks later? A At the time the work was completed.

Q The dates were kept as you have testified about the dates in the records? A Yes.

BY THE COURT:

Q What is the significance of the asterisk on Exhibit 27, opposite 601-321A? A I don't believe that has any particular significance, Your Honor. That is our way to call to our attention a particular repeat test.

62 Q I notice that there is a blank line throughout these exhibits between each test, except with reference to 601-321, on Exhibit No. 27, and 321A. Does that have any significance? A The only significance of that is this: When we set up composition 601-321, we didn't anticipate a repeat test and didn't provide a section below that for recording a repeat test.

It has been our practice, as far as we are able, to keep repeat tests as close to each other as possible for convenience in checking back on our records.

So when the test on 601-321 was completed on 11-3-41, we made our entry, and decided to repeat that particular test, and we decided to make the entry as close as possible to 601-321, just for convenience. We didn't have much room but we squeezed that particular sample in right below that.

BY MR. LIMBACH:

Q And that might explain why that test is apparently out of its chronological order; is that right? A Yes.

You see, the A numbers can appear anywhere throughout the record, and it is arranged to have them as close together as possible and not scattered all over the code book. This makes for convenience in checking back on the records.

BY THE COURT:

63 Q Was that done in other places in your records? A Let me see. I haven't paid particular

attention to that. I am going through the record to see if I can find an example like that.

MR. SCHIMMEL: You might check Exhibit 23 and 24, Your Honor, on 601-302 and 601-302A appearing on Exhibit 24.

THE WITNESS: Although they are not right underneath each other, they are as close as the record would permit us to keep them.

BY THE COURT:

Q What exhibit are you referring to? A Exhibit 23 and 24 of the exhibits he made reference to.

Sample 601-302 appears on Exhibit 23, and Exhibit 24 being the next page in our code book, and the sequence of numbers on the bottom of the page on Exhibit 23 is 601-304, and at the top of Exhibit 24 is 601-305, so that would be the next page in our code book, and a repeat on 601-302A appears on that particular page.

Q It appears in chronological form according to dates? A Yes, sir.

Have I explained the original question, Your Honor?

THE COURT: Yes.

MR. LIMBACH: Shall I proceed, Your Honor?

THE COURT: You may.

64 BY MR. LIMBACH:

Q I hand you a soft copy of your patent No. 2,261,047, based on application for patent serial No. 404,361, filed July 28, 1941, which is in the record.

MR. SCHIMMEL: That is A in the folder, Your Honor.

BY MR. LIMBACH:

Q I note in the specification of your patent there are used many terms which you have used in your testimony. There are given certain preparations, and what I would like to ask now is whether the various terminology used in this patent is consistent with the terminology you have used in your testimony in this present case? A I believe that is correct.

Q Do you identify there a material which you testified about as being ZMP? A Yes.

Q What is that material again? A Zinc salt of methyl cyclohexanol and P2S5 reaction product.

Q And is the material which is identified in the claims of that patent the same ZMP material? A That is correct.

THE COURT: At this point I am going to ask the marshal to announce a 10-minute recess.

(Thereupon a short recess was had.)

65 BY MR. LIMBACH:

Q I should like, Doctor, to make sure that we have in the record your testimony as to the date on which the composition No. 6 was tested in the Lauson engines. A Referring back to code No. 601-313—

THE COURT (interposing): The witness testified that the test started September 25, 1941, and was completed on October 3, 1941.

BY MR. LIMBACH:

Q I have one further question. Your patent application involved in this proceeding is limited to the use of zinc as the metal in preparing the salt? A Yes, sir.

Q Why was it limited to the specific metal? A You mean the metal, pertaining to the P2S5, or alcohol reaction product?

Q That is right. A We were interested in a compound which was substantially soluble in all mineral oils, rather than one having limited solubility.

Q Do you have any record to show that by the use of other metals a product not complete soluble was produced? A Yes.

Referring to Exhibit No. 23, under code 601-304, which
is the last sample on that page, it describes a ma-
66 terial which is a reaction product of capryl alcohol,
5 mol, and 1 mol of P2S5, made into calcium salt;
prepared by double D composition from sodium salt.

On Exhibit 24, under sample No. 601-305, at the top of the page, and sample 306, and next to that is 307, which shows we attempted to prepare a composition of oil, plus this sample 601-304 in a 2 per cent concentration, and had to omit the test because the material was insoluble, and by that I mean, it wasn't completely soluble in the oil media.

MR. LIMBACH: You may cross examine.

Cross Examination

BY MR. SCHIMMEL:

Q I think to cut through a lot of exhibits here, can you point out which exhibits are those relating to what you are claiming in this particular application, and eliminating everything that is not being claimed? A Under Example 6, we find a composition which is being claimed.

Q On Exhibit 6 is which? A Example 6, in reference to Exhibit No. 25, 23, 11, 36, and 40.

Q Which exhibit shows the preparation of the zinc capryl alcohol dithiophosphate? A Let me check that. That is Exhibit No. 23, I think, under sample number, code number 601-302.

67 Q That is the first time at which that particular compound was made according to these exhibits? A That is correct.

Q Is that correct? A Yes, sir.

Q I would like to call your attention to claim 1 in Plaintiffs' Exhibit 43, which was the first claim, and ask you to read that claim.

It is the first claim in the bill, Your Honor.

A Read the claim?

Q Yes. A A lubricating composition containing a characterizing amount of the product obtained by the simultaneous admixture of capryl alcohol, phosphorus pentasulphide, and zinc oxide.

Q I direct your attention particularly to the word "simultaneous admixture", two words.

Do you have any exhibit which shows a preparation as called for in that claim? A Let me refer back to 601-302, under Exhibit 23, and see what that says.

I don't find anything that is strictly simultaneous.

Q In other words, there is no preparation of the zinc,
capryl alcohol, phosphorus materials simultaneously
68 admixed that you can find A No, I cannot find
any.

Q This exhibit, code number 601-302, on Exhibit No. 23, you testified, I believe, it was not made by you? A That is correct.

Q It was made by someone else? A This was entered by someone else.

Q Was it prepared by someone else? A I don't recall exactly, but I believe it could have been. The person who entered this was in charge of the organic group.

Q Now, in connection with the typewritten Example No. 7 which you have testified about and for which you say that there was an engine test run, according to the sheet there, you did testify that it was similar to the—I mean, referring to page 8A of Plaintiffs' Exhibit 43, the original application—you testified that it was similar to, as I recall, lubricant No. 6 in this table A.

Now, on this Example 7 I note you have 16 per cent of calcium petroleum sulphonate, and in the application you have 1.6 per cent of calcium salt of petroleum sulphonic acid.

Would you say these were the same?

THE COURT: Would you repeat the last part of that question?

BY MR. SCHIMMEL:

Q In the application you have 1.6 per cent of
69 calcium salt of petroleum sulphonic acid.

My question is: Would you call those similar? A The proportions present in both samples are the same. The concentrations of these ingredients are different.

This is an oil blend for purpose of engine test. This is a concentrated solution of those ingredients, such as when you use 10 per cent by volume you would obtain these proportions.

Q In Example 7 that is not a lubricant that would be used in an engine? A That is correct.

Q In your application, this would be used? A Yes, sir.

Q As far as 7 is concerned, that was never used as such in an engine? A That is correct.

Q I would like to refer to Exhibit 40 for a moment, and I think you testified that the date typewritten in the right-hand corner of that exhibit, which appears as 9-25-41, was the summary of the data after the tests were made? Is that correct? A That is correct. That is a continuous summary of the engine test.

Q That is a summary of the engine tests? A Yes, sir.

70 Q And presumably engine tests have been made before that date? A Well, the records which appear on this sheet do not necessarily mean that they were all done by 9-25-41 because this table could be added to from time to time as engine tests were completed.

If the tests weren't completed on the date, and we needed a summary of the tests which had been completed, and we show them all on one page, the tests could be typed into that summary from time to time.

Q And the date remained the same? A Well, the date would signify the date on which the summary sheets were started or initiated.

Q Then it is the date on which the start was made? A That is correct.

MR. SCHIMMEL: I merely call that to the Court's attention because of the fact that on Exhibit 26 in the test of 601-318, apparently the completion date is 10-10-41, and yet apparently the summary was made before the test was completed.

THE WITNESS: The summary could not have been made before the test was completed. The summary is a continuous record of the completed engine test, and that date at the top of the page signifies the start of that particular page of the summary.

71 MR. SCHIMMEL: That is all I have, Your Honor, at the moment.

BY MR. LIMBACH:

Q The date which establishes the date on which the engine test is run, according to your direct examination, is the date on the code sheet? A That is correct. It is the code sheet of original entry that establishes the absolute date of the start and the finish of the engine test.

MR. LIMBACH: I have no further questions, Your Honor.

THE COURT: You may step down.

(Thereupon the witness was excused and retired from the witness stand.)

MR. LIMBACH: I would like to call Dr. Kelvin Smith as my next witness.

Thereupon,

Kelvin Smith

was called as a witness by the plaintiffs and, being first duly sworn, was examined and testified as follows:

Direct Examination

BY MR. LIMBACH:

Q Will you please state your full name and your residence address? A Albert Kelvin Smith. My residence address is 22099 Shaker Boulevard, Shaker Heights, Ohio.

72 I would like to add that I have dropped the Albert from my name because of confusion in my family. Now I just use the name Kelvin Smith.

Q What are your academic accomplishments by way of degrees, Dr. Smith? A I have an A.B. degree from Dartmouth College.

A B.S. degree in chemical engineering from Case School of Applied Science, which has changed its name since I got my degree to Case Institute of Technology.

I also have an honorary degree of doctor of science from the same institution.

Q What is your occupation, Dr. Smith? A Chemical engineer.

Q Connected with what company? A The Lubrizol Corporation, where I am vice president in charge of research and development.

Q Are you familiar with the Asseff application involved in this proceeding? A Yes, sir.

Q And the invention therein disclosed in the findings? A Yes, sir.

Q I would like to have you summarize for us the research program leading up to and including the invention which is claimed in this Asseff patent application.

A The particular objective in this line of research,
73 which includes the development involved here, was
to produce a concentrate which could be added in minor proportions to a motor oil to produce a crankcase lubricant which would satisfactorily lubricate a heavy-duty internal combustion engine, and particularly high-speed Diesel engines, which had so-called corrosion sensitive bearings; that is, a final lubricant prepared by blending a small proportion of the concentrate with the base oil, which should have both detergent properties, in order to minimize the deposits in the heavy-duty engine, and corrosion inhibiting properties to protect the bearings from the products of decomposition of the oil and the fuel.

That was a difficult objective to reach because it had been found by earlier workers that in general the things which had detergent properties were corrosive to corrosion sensitive bearings, and the things which were corrosion inhibitors acted in general to form coatings on bearings, and would therefore be overcome in their action by detergent materials which would presumably remove the coating.

Q Will you proceed?

I asked for a more or less chronological history of the development program leading up to and including the
invention here in issue. A During the period from 1938
to 1940, there was a great demand for detergent ad-
74 ditives because of the growth of the high-speed
Diesel engines, which would not operate satisfac-
torily with untreated oils (that is, mineral oils which did not contain chemical additives), because of the fact that deposits form so readily in the combustion chamber that the engines could only be operated for a short period of time, and then they would have to be taken down and cleaned out.

The first materials which were successful in that field of use were oil soluble soaps, principally, and which were quite effective in reducing deposits formed in the combustion chamber.

The success of those materials led to longer periods of operation in general between over-all periods and revealed that the bearing materials, which were principally babbitt at that time, were not satisfactory for such long sustained periods of operation and resulted in the development of higher melting point and stronger, mechanically stronger bearing material, such as lead-bronze, and these in turn proved to be particularly sensitive to corrosion from the products of decomposition of the oil, and to the additives themselves which had been used as detergents up to that time.

It was at that point that the need for a crankcase lubricant having the combined properties of a high degree of detergency along with bearing corrosion came about—no, with bearing-corrosion inhibition, I should have said, came about.

75 Q I hand you a soft copy of Patent No. 2,261,047,
of record in this proceeding, and ask you if it refers

to one phase of the development program in your company about which you have been testifying? A Yes, it does.

Q Will you tell us something of the nature of that material, and the nature of the material and the use of which in lubricants is covered by the claims of that patent, and the material which is covered by the claims of the patent application involved in this proceeding? A The development of this material, the zinc salt of the reaction product of phosphorus pentasulphide with alcohol, which is the subject matter of this patent, in a crankcase lubricant was an important step in that development because of the combined characteristics of a very effective corrosion inhibitor with the property of not being overcome in its effectiveness by the presence of effective detergents.

In other words, it was a material, a very effective corrosion inhibitor, which could be used with effective detergents to produce a kind of combination that had been sought for the purpose I have described.

Q Now, its relation to the material which forms the subject matter of the claims in issue here. A The difference between the material covered by this patent
and the characterizing material in the patent
76 application with which we are concerned in this
case is found only in the alcohol which is used to
form the reaction product with phosphorus pentasulphide, and then neutralized with zinc oxide to form the zinc salt.

In other words, both materials are zinc salts of the reaction product of an alcohol with the phosphorus pentasulphide. In the case of Patent No. 2,261,047 the alcohol is methyl cyclohexanol, in the application at issue here the alcohol is capryl alcohol.

Q Has your company ever commercialized the material of Patent 2,261,047? A Yes, it has.

Q Has your company ever commercialized the material that forms the subject matter of the claims here in issue? A Yes, it has also.

Q I note that the application on which Patent No. 2,261,047 is based was filed July 28, 1941, and issued fairly promptly, namely, on October 28, 1941.

Do you recall or were you familiar with the circumstances surrounding the prosecution of that patent application? A I do recall the circumstances and I am familiar with them.

Q What were they and how were they related to your
commercial program? A The laboratory work
77 had been completed some time prior to the filing of
this application.

THE COURT: The witness is now referring to 2,261,047?

MR. LIMBACH: Yes, Your Honor.

BY MR. LIMBACH:

Q Proceed. A And the pilot plant work had been completed also, so that the product was ready for manufacture and sale.

In view of the considerable commercial demand for a product of this kind, the Lubrizol Corporation was anxious to put the material on the market to supply that demand. For the company's protection that led us to ask to have this application to be made special so it could be issued into a patent at the earliest possible date in order to protect the commercial development of the product.

Q You say, however, not only was the material of the patent 2,261,047 sold, but your company also sold the material covered by the claims of the present application; is that right? A That is correct.

MR. LIMBACH: On this point I would like to state that one of the or, rather, the principal reference apparently relied upon by Mr. Schimmel is the Cook patent.

Now, the Cook patent was filed in November, and the application on which it is based was filed on November 8,
1941. That is Patent No. 2,344,392.

78 Mr. Schimmel in his opening statement yesterday
gave as the effective date of that Cook patent as

July 11, 1941, which is the filing date of Serial No. 401,960, referred to in the concluding paragraph of the specifications of the Cook Patent No. 2,344,392.

Included in the file of the Asseff patent here involved, as Exhibit 43, is an abstract of the Cook application, Serial No. 401,960, beginning on page 95 of Exhibit 43. I shall rely on that portion of Exhibit 43 to show, first, the extent of the disclosure of that Cook application, 401,960, filed July 11, 1941, as well as certain statements made in the application which are inconsistent with statements made in the application which matured into Patent No. 2,344,392.

Patent No. 2,344,392 is endorsed on its face that it is assigned to the American Cyanimid Company of New York.

BY MR. LIMBACH:

Q And I now ask the witness whether he knows of that company, particularly with regard to it being a competitor of the Lubrizol Corporation in the sale of materials like those with which we are concerned. A Yes. The American Cyanimid Company is a competitor of the Lubrizol Corporation in the field of manufacture and sale of lubricant additives, and particularly this class of material.

MR. LIMBACH: Now, Your Honor, Dr. Smith
79 has identified himself as being director of research of the Lubrizol Corporation. The objective of my further questioning and proof of my further questioning would be for him to corroborate the exhibits and documentary evidence that the first witness has testified about. I can do that in much the same way as by the testimony of the first witness, or I would like to ask Mr. Schimmel whether he is willing to stipulate that.

MR. SCHIMMEL: I have no objection to that. I think we can save a lot of time.

THE COURT: The stipulation which you will enter is that this witness would corroborate the contents and the material, and the dates and the nature of the tests, and that the entries were made as testified by the first witness?

MR. SCHIMMEL: I should say I will go so far, Your Honor.

THE COURT: Does that serve your purpose?

MR. LIMBACH: Yes, Your Honor. It saves me drawing a stipulation.

I would like to have one further question on that point, however.

BY MR. LIMBACH:

Q With regard to the direction and control of the
program, how is that delegated in your organiza-
80 tion, Dr. Smith? A The program of the research
work—

Q (Interposing) In other words, how can it be explained that this application was filed in the name of Dr. Asseff, whereas on direct examination he stated that certain entries with regard to one or two of the preparations were made by, as I recall, Dr. Drake? A The program of the research work is under my direction and is discussed with the various members of the research laboratory in terms of general directions, that is, we decide to make certain types of materials to accomplish certain objectives.

Then the particular detail of how that program is carried out is in the hands of all the members of the research department, of which Dr. Asseff is one, and as that program progresses, ideas occur to the other members of the organization and are communicated to me.

Now, the particular detail of how these ideas are carried out then depends on the nature of the operation and are assigned to the workers who are skilled in that particular operation involved.

Q Do you recall who initiated or originally proposed the invention which is the subject matter of this proceeding? A Yes, I do—Dr. Asseff.

Q What was then done by way of carrying out the reduction to practice, the testing out of that idea?

81 A Dr. Asseff outlined the particular compounds to be prepared for this purpose, and they were then, the preparation of these compounds was then assigned to the laboratory workers skilled in that type of operation.

MR. LIMBACH: There was one exhibit in the booklet which I inadvertently failed to have Dr. Asseff testify about, and that is Exhibit No. 36. I should like to ask Dr. Smith if he knows, and I might mention, Your Honor, that the page is not numbered in the book but it is in your book, but it is not numbered. It is the blueprint.

THE COURT: That is the soap exhibit?

MR. LIMBACH: No, this is another one. This is 36—no, 39, I am sorry. I forgot to put the number at the bottom of the sheet, that is all. That should be numbered 39.

THE COURT: There was testimony about this exhibit, was there?

MR. LIMBACH: None so far.

BY MR. LIMBACH:

Q I now ask you, Dr. Smith, if you know what that exhibit is and what is shows? A This is a report of the result of a Chevrolet engine test. The particular procedure in our laboratory and other laboratories is identified as the L-4 procedure, which is a high-speed, 82 relatively high load continuous test of 36 hours in the engine from a Chevrolet automobile, by means of which the suitability of the crankcase lubricant is tested for that type of service.

The engine is inspected at the end of a test for deposits, and those appear in this report in two categories, one, in varnish deposits, and 10, a rating of 10 for each of the individual items under varnish deposits, indicates that the part inspected is free from deposit.

Then as the number is less than 10, the difference between 10 and the number noted here indicates that there are increasing amounts of deposit as that difference increases.

For example, 9.5 rating means that there is very little deposit, and if there were a large amount of deposit, the rating might be, say, 5.

Now, those ratings, as you can see, are then taken to determine the overall varnish deposit, and that is given on the basis of 100. 100 is perfect, indicating one with no varnish deposit—that is not correct—50 in this case would be for the varnish deposit, that would be a perfect rating.

And the same thing is done in sludge deposits, as shown in the column there; and there again, if the individual items inspected had a perfect rating of 10, the perfect rating at each of these five inspected points would be
50, and the overall rating is determined by adding
83 the total varnish rating to the total sludge rating,
and gives the figure shown, the combined engine deposit rating of the varnish and sludge, where a perfect rating would be 100.

The other characteristic of the oil, the crankcase oil, tested by this means is the bearing corrosion effect, which is noted under copper-lead bearings on this report sheet. It is reported in two ways, first, as the loss in weight of each half of two different bearings, the top half and the bottom half, in terms of grams, and also by observation as indicated by the notation under the loss in weight, which says in this case, "fair, slightly rough, no varnish" —a satisfactory non-corrosive oil.

This test should show an average loss in weight for each whole bearing of about between 200 and 300 milligrams, or between .2 and .3 grams, depending in part on the appearance of the bearings as well. In other words, Your Honor, if it didn't have a satisfactory appearance, that weight would have to be lowered in order to constitute a satisfactory result.

Q There are two sheets which comprise Exhibit 39. I was referring first to the one identified in the upper right-hand corner as Test No. 1-601-4875. A I would like to say that my comments have been directed to what appeared on that sheet—up to this point.

84 MR. LIMBACH: This is the original, Your Honor.

THE COURT: I have followed the copy that I have.

BY MR. LIMBACH:

Q Will you tell us about what the material was that was tested and the results secured with regard to that material? A Yes.

The material tested, the lubricating composition tested is shown opposite the place on the sheet that says, designation of oil, where it says or it is identified as Sun 90VI.

Q Will you explain those terms as you go along, please? A That is a mineral oil supplied by Sun Oil Company as a motor oil, and its 90 viscosity index is indicated by 90VI, which means that it is near the top of the scale used to show the rate of change of viscosity with change in temperature for oils in that class of service.

100VI would be an oil that has a very low rate of change with change in temperature as compared with the average oil used for that purpose.

The 20 per cent Bright Stock indicates this particular base oil is a blend as is usual in that type of product of a relatively light oil, which is usually called neutral, and a relatively viscous oil, which is called Bright Stock,
85 and the two being blended together to furnish the
proper viscosity for a given type of service.

That oil had in it .81 per cent of material shown here as ZnCapMP, which identifies the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide.

Q Is that a material like that called for by the claims in issue here? A Yes, it is.

As I have already indicated, the test results shown on this sheet would be considered as very satisfactory.

In other words, this oil, both as to deposit ratings and effect on the bearings, is an excellent crankcase lubricant as indicated by this test.

Q Would you now refer to the second sheet, of Exhibit 39, please, and tell us what it shows. A It is a test run under the same procedure in which the base oil is the same as that previously described, Sun 90VI, and the oil contained .81 per cent of a material indicated here as ZnCetMP, which would identify the zinc salt of the reaction product of cetyl alcohol with phosphorus pentasulphide.

Q What is the difference between cetyl alcohol and capryl alcohol? A They are both aliphatic alcohols. In
other words, the carbon atoms are arranged in a
86 chain; but they differ in two respects. Capryl al-
cohol contains only 8 carbon atoms in the molecule,
while cetyl alcohol contains 18.

They differ also in the point at which the hydroxyl group which characterizes an alcohol, is attached to the carbon chain. In the case of capryl, the hydroxyl group is attached to the second carbon atom from the end of the chain, the hydroxyl group, while in the case of cetyl alcohol it is attached to the end carbon in the chain.

Q Did you refer to the results of that test? A I haven't yet.

The deposit, the total deposit rating is not significantly different from that shown on the previous test for the capryl alcohol product, but the amount of bearing corrosion is very greatly different.

In this case the total loss in weight per bearing is in excess of 2 grams, and the bearing has been observed to be, according to the notation here: poor, rough, and with no varnish deposit on it.

And the amount of loss in weight of the bearing would show the removal of so much metal from the surface as to seriously damage the bearing and make it unsatisfactory for further use.

Q Were you familiar with these tests at the
87 time they were run? A Yes.

Q Were you familiar with the procedures followed in these tests to know whether or not they were standard procedures, identical in both tests? A Yes, the procedure is standard in that it has been adopted by the Army Ordnance Department in a specification for heavy-duty motor oil for use in mechanical equipment of the armed services.

Q And that was followed in both tests? A Yes, sir.

Q And the same conditions? A That is correct.

MR. LIMBACH: Now, Your Honor, I have one further line of questions for this witness, and that is under the authority of the decision of the Court of Appeals for the District of Columbia in the Knutson v. Gallsworthy case, 164 Fed. (2d) 509 to which I referred yesterday, where the Court indicated the desire for technical assistance in the interpretation of patents which they might be called upon to consider from the standpoint of their anticipatory effect.

I think Dr. Smith is eminently qualified as a chemist and skilled in this particular art to review the prior art which is in issue and comment thereon from the standpoint of the chemicals therein disclosed, so that we may have in the record a basis for ready comparison
88 between the prior art and what is here explained.

THE COURT: Do you question the Doctor's qualifications in this regard?

MR. SCHIMMEL: No question at all, Your Honor.

THE COURT: Very well. The Court will then hear the testimony.

I think, however, that we will take a short recess at this time before we start this additional testimony.

(Thereupon a short recess was had.)

MR. LIMBACH: With regard to the Cook patent, No. 2,344,392, I believe it unnecessary to question the witness because that happens to be antedated.

As to the remaining references relied upon by Mr. Schimmel—would you care to indicate which ones they might be, Mr. Schimmel? I understand you are not going to rely on the Musselman?

MR. SCHIMMEL: I didn't mean Musselman. That got in by mistake. D on the folder can be scratched out.

The only reason Asseff, A in the folder, is in there is because of the specific mineral oil used.

In Claim 5, for example, there is a particular designation of the viscosity number, and so forth, and that is the only reason that is there, to show that has been used before.

The other references, McNab, Salzberg, and Wilson are relied upon. Salzberg and Wilson for their dis-
89 closures of thiophosphate esters and metal salts of
thiophosphate esters as lubricant oil addition agents, as well as the fact that they disclose using metal phenates as additional additives.

Wilson also discloses the sulphonate salts as additional agents to be used in conjunction with thiophosphor esters.

McNab also has a disclosure of using other corrosion inhibitors in conjunction with thiophosphate ester salts. Those, I believe, are what I will rely on in these patents.

MR. LIMBACH: I see. We will take them up in the order in which they are set out in the amended answer in which Salzberg 2,063,629 is first.

BY MR. LIMBACH:

Q I hand you a copy of that patent, Dr. Smith, and I would like to have you tell us what you consider to be the nature of the chemical compounds to which that patent is directed. A By looking at the claims, I find that this patent is concerning an aliphatic ester of thio-acids of phosphorus, in which at least one aliphatic radical contains at least 10 carbon atoms.

Now, one method of preparation is given for one class of such esters by reacting—

Q (Interposing) In referring to the patent, would

you tell us where you find the things that you refer
90 to, please? A All right.

—an alcohol having a carbon chain of at least 10 carbon atoms with phosphorus pentasulphide.

This is disclosed, for example, in Example 1 where the preparation of the ester is accomplished by reacting dodecyl alcohol with phosphorus pentasulphide, dodecul alcohol being an aliphatic alcohol that contains 12 carbon atoms in a chain.

Q I refer you particularly to the first paragraph, page 1, column 2, that is presumably the basis for the limitation in his claims that the esters must contain at least one aliphatic chain of at least 10 carbon atoms; is that right? A That is correct.

Q What is the difference between that compound, or those compounds, and the compound to which the claims here in issue are directed, namely, the zinc salt of the reaction product of capryl alcohol and P2S5? A Capryl alcohol does not contain a chain of carbon atoms in which the number of carbon atoms is at least 10. It only has 8 carbon atoms in the molecule altogether.

Q Do you find in the Salzberg patent any compound identified as a zinc salt of the esters with which Salzberg
is concerned? A Yes. On page 3, the second
91 column, line 17, there is a paragraph which says
that the invention embraces the acid esters and their salts.

In other words, he says that salts may be made from the acid esters and says that his invention embraces such salts.

Q And is zinc mentioned in the list of metals? A Yes, it is in line 19.

Q But does he identify any specific compound which is zinc salt of the esters of his invention, and does he give any method of preparing such zinc salts? A No, he does not.

THE COURT: Will you read the last two questions and answers?

(The last two questions and answers were read by the reporter.)

BY MR. SCHIMMEL:

Q Is that "No" in answer to both questions? A He does not. Shall I clarify it?

BY MR. LIMBACH:

Q Will you please? A He does not identify any specific salt, nor does he give any method of preparation of the salt.

Q And since his patent claims these materials as new compositions of material, in other words, he was the first to have prepared these materials, certainly there was no
earlier teaching anywhere on how to make these
92 zinc salts; is that right? A Yes.

Q So much for Salzberg.

The next patent is Wilson, No. 2,280,419.

I hand you a copy of that patent and ask whether you are familiar with it. A Yes, I am.

Q What is the invention to which that patent is directed? A By reference to Claim 1, I find that the invention claimed is a mineral lubricating oil containing a small proportion of an oil-soluble petroleum sulphonate and a small proportion of the salt of a weak non-carboxylic organic acid material having an ionization constant not exceeding about 5 times 10 to the minus 6 that is claimed.

Q For purpose of convenience, I shall refer to the salt compound mentioned in the claim as the second component. Is that second component like the zinc salts of the claims here in issue? A No, it is not.

As indicated by examples in the specification of the kind of materials described as the second ingredient, the second additive ingredient, those are phenolic materials, and the phenols are very weak acids, which fall within the range
of the ionization constants given at the end of the
93 claim which I have just read, while the reaction
product of capryl alcohol with phosphorus penta-

sulphide is a strong acid having an ionization constant much higher than that given in this claim; for an acid having strength comparable with the ordinary inorganic or mineral acids. It would be far outside of the range indicated by the ionization constants given in this claim.

Q Would it be proper to designate the zinc salt of the reaction product of capryl alcohol with P2S5, or at least a major portion of such reaction product or salt, as a dithiophosphate? A Yes.

Q Do you find any dithiophosphates disclosed in the Wilson patent? A It would take quite a while to answer. I have examined the section which deals with the constituents, which is the second one or, rather, the last one in Claim 1, and there would be no examples of that, and I know there are no examples of dithiophosphate in that section of the specification.

Do you want me to go through the rest of it and look for it?

Q You might. Would you, please.

94 It won't take very long, Your Honor. A No, I don't find any dithiophosphate in the patent.

Q The next patent is McNab, No. 2,289,795, of which I hand you a soft copy and ask you what is the invention to which that patent is directed? A By reference to Claim 1, I find that this patent is concerned with a mineral lubricating oil which contains small amounts of polyvalent metal salts, selected from each of the groups consisting of aluminum, and alkaline earth metal salts, the total amount of such metal salts amounting to from .1 per cent to 3.5 per cent by weight of the oil and the amount of each type of metal salt being at least one-tenth of the total metal salt present.

Q I direct your attention particularly to page 3, column 2, line 70. A Yes.

Q And ask you what the compound is which is there disclosed? A Under the head of thio-phosphates, there is a compound calcium dioctyl thiophosphate.

Q How does that compound differ from the characterizing compound of the compositions covered by the claims in issue here? A In two ways. First, a different metal is used to form the salts. In this patent it is calcium,
rather than zinc as in the Asseff application, and
95 the other way is that the alcohol used to make the
dithiophosphate ester is the octyl alcohol.

THE COURT: Is what?

THE WITNESS: Is the octyl alcohol, which in the absence of any prefix is presumably normal octyl alcohol.

Whereas in the Asseff application the alcohol is identified as capryl, which is a secondary alcohol.

MR. LIMBACH: Those seem to be the only three patents not previously mentioned. Those are the only three patents whose disclosure may require interpretation.

You may cross examine.

Before I complete my examination, I should like to offer in evidence all of the exhibits which have been thus far marked for purpose of identification, as Plaintiffs' Exhibits No. 1 to 47.

You may cross examine.

MR. SCHIMMEL: Shall I interpose my objection to these now, Your Honor, or continue with the cross examination and then take up the question of urging those be not admitted?

THE COURT: The Court has no preference. Whichever counsel desires to present it.

MR. SCHIMMEL: Does it make any difference to you?

MR. LIMBACH: No.

MR. SCHIMMEL: Suppose we have the cross examination first.

96 *Cross Examination*

BY MR. SCHIMMEL:

Q Dr. Smith, in Exhibit 39 which you described, in the designation of the oil component used in those tests, you said that it contained .81 per cent of something that carpared? A That is the usual way of trying, if a strong acid is used.

Q But you have testified that this was a strong acid? A Yes.

Q So you would expect that to react with the alkaline material as a salt? A I have learned that. I didn't know that was available knowledge at the time of this patent.

Q It is available knowledge with respect to very strong acids? A Yes. I have learned that is a strong acid. That wasn't available knowledge, I don't believe, at the time this was published.

Q It would be available to any chemist on hydrogen ionization? A It is a difficult thing to do.

Q Couldn't they determine it through their experience within hydrogen ionization, for example? A I can only answer that that is a difficult measurement to make with this type of material.

It is ordinarily done with water soluble materials, but in a case where it is not water soluble, it would be difficult.

102 Q Some of these would be water soluble? A No, sir. As far as I know, none of them.

Q None of them? A As far as I know, no. None of the acids, which is what we are talking about.

Q I would like to call your attention to the same page of the Salzberg patent, beginning with line 37. A The same column?

Q The same column, yes, to line 42.

The patentee states that these compounds of our invention may be employed as lubricants for bearings.

And also, line 67 of the same column, in which he characterizes those as extreme pressure lubricants.

Let us turn to—

MR. LIMBACH: Is that a question?

MR. SCHIMMEL: I am just calling his attention to it.

THE COURT: There is no question before the witness.

MR. LIMBACH: I am sorry.

BY MR. SCHIMMEL:

Q Turning to McNab for the moment, which is C in the folder, you testified as to the compound on line 70, on page 3 of that patent, that it differed from the compound specifically claimed in the pending case in two particulars, first, that it was a calcium metal instead of zinc metal, and second, that it used an octyl alcohol instead of a capryl alcohol.

103 Now, isn't capryl alcohol an octyl alcohol? A Yes. Octyl means eight. They both have eight carbons.

Q Dr. Asseff testified, and in going through these typewritten examples, Example 6 compared with Example 8, and as I recall his testimony it was that they were very similar to each other.

Could you check that? A Perhaps if I might have that testimony read to me, I would be able to do it.

Q As I recall the language, they were similar to each other. A I don't believe so.

MR. LIMBACH: I would like to have the testimony read back.

THE COURT: The witness testified that Examples 6 and 8 were similar, except for the alcohol used in preparing the two compounds. One was capryl alcohol and the other octyl alcohol.

MR. SCHIMMEL: He used the word "similar"; is that right?

THE COURT: The witness used the word "similar," except for the alcohol.

MR. SCHIMMEL: I remember that now.

BY MR. SCHIMMEL:

104 Q You agree with that, that they are similar? A Yes; in exactly the way Dr. Asseff explained it.

Q How would you characterize the similarity, in what sense? A For the same pair of alcohols I have referred, as between the compound disclosed in the McNab patent and the compound disclosed in the Asseff application in this case, in one case it is normal octyl alcohol, which is a primary alcohol, where the hydroxyl group, which is the characterizing group of the alcohol, is attached to the end carbon of the chain. That is the type alcohol used in Example 8.

Capryl alcohol is a secondary alcohol, where the characterizing hydroxyl group is attached to the second carbon atom from the end of the chain, a change which gives an alcohol different characteristics.

Q Are they similar in functions? A The word function means nothing to me there.

Q Are they similar in the use to which they were put, as far as these examples were concerned? A The hydroxyl group is the one that takes part during the reaction with the acidic material, such as phosphorus pentasulphide.

Q In use as a lubricant additive, they are similar? A The additives resulting from the acids that are prepared from the two different alcohols were tested in
105 Example 6 and 8 in exactly the same way.

Q And both found to be O. K., as far as you know? A Yes.

THE COURT: I do not think the previous question was answered. Read the last two questions and answers.

(The last two questions and answers were read by the reporter.)

THE WITNESS: They are similarly used in these two experiments.

BY MR. SCHIMMEL:

Q And they would be found similarly effective? A I would have to refer to the engine test for that question.

MR. LIMBACH: The data with regard to the test is of record, and that is the first evidence, Your Honor.

THE COURT: The witness may refresh his recollecries this designation, ZnCapMP, and you interpreted that to mean it was the reaction product of zinc capryl alcohol and phosphorus pentasulphide.

Is there anything in these exhibits which would show that to be so? A I don't believe so.

MR. SCHIMMEL: That is all as far as that is concerned.

Do you have copies of those references?

MR. LIMBACH: Yes, I do.

MR. SCHIMMEL: Do you want to give them to him again?

MR. LIMBACH: McNab, Wilson, and Salzberg?

MR. SCHIMMEL: Yes.

MR. LIMBACH: Do you want the witness to have them?

MR. SCHIMMEL: Yes, if you don't mind.

BY MR. SCHIMMEL:

Q Take the Salzberg patent, and on page 1 of that patent, the second column, line 3, the patentee states he uses aliphatic alcohol; is that correct? A Yes.

97 Q Is capryl alcohol an aliphatic alcohol? A Yes.

Q Then on page 3. You stated that though the patentee stated that his invention embraces the salts of the particular esters specified, and you agreed that he mentioned the zinc salt? A Yes.

Q You stated that there was no specific zinc salt mentioned; is that correct? A That is correct.

Q The claim in this case, if you recall, calls for the reaction product of three materials. A Yes.

Q Does that specify any particular material? A Yes.

Q A reaction product of zinc oxide, phosphorus pentasulphide, and capryl alcohol specifies what specific material? A It specifies all those materials, specifies materials needed to give an end product.

Q But no specific product? A I don't understand the question.

Q Put it this way: Do these three materials when reacted produce one single material? A Yes.

Q Under all circumstances, in all proportions?

98 A If you say single material—there may be small proportions of more than the principal compound produced.

Is that your question?

Q Well, does the claim call for a principal compound? Can you designate what compound the claim calls for? A Yes, I know what the principal reaction product will be when those three things react.

Q Perhaps I should show you the claim.

I call your attention to Claim 1, which specifies the product obtained by the simultaneous admixture of capryl alcohol, phosphorus pentasulphide, and zinc oxide.

Does that claim specify a particular compound or can it cover a number of compounds? A Your question is not clear to me. If I understand what you mean, I think I can answer it by saying that when these materials, the three materials react, a major proportion or nearly all of the resulting reaction product will be a single compound, which is a single specific compound.

Q But there are other compounds produced as well in that reaction? A If at all, only in relatively small proportions.

Q Does that always hold true regardless of the relative proportions of the material used in making the product? A Yes, as far as I know.

99 Q The same single compound that you speak of would be produced? A Yes. The excess of any one or more of the products there reacted with the others would appear in the end product as a separate material. So that if you are talking about the reaction product, it would essentially be a single compound.

Q May I ask what would that product be? A It would be the zinc salt of dicapryl dithiophosphate.

Q That would be true regardless of the proportion of the capryl alcohol you started with? A As far as I know.

Q Do you know what proportion was used in the actual exhibits on file here? A Yes.

Q What was it? A There were 5 mol of alcohol to 1 mol of phosphorus pentasulphide.

Q Getting back to Salzberg again, page 3 of the patent, where he speaks of making his zinc salts.

THE COURT: Where is that?

MR. SCHIMMEL: Lines 17 to 20, Your Honor.

THE COURT: All right.

BY MR. SCHIMMEL:

Q That method was known of making those salts
100 before Mr. Salzberg filed his application? A My
only knowledge of the method required to produce such salts had been gained by actual experience with the materials referred to here as having been prepared in our own laboratory.

Q You know of no way of making salts, except through your own experimentation? A The specific salts, that is what the question was, I believe.

Q Salts of these, of your own thio esters? A Yes.

Q Was the method of making them developed in your laboratory? A Yes.

Q They weren't known before? A I don't know that. I said my knowledge has been gained from actual experience with the materials made in our own laboratory.

Q Well, as a chemist wouldn't you expect to make a salt in the normal manner of making a salt, which is to add alkaline material to the acidic material and heating them under reaction conditions? A Sometimes that works; sometimes it doesn't.

Q As a general rule it works, doesn't it? A I
can give you a very large number of examples
101 where it doesn't work.

Q Isn't that the way generally salts are pretion from an examination of the test in order to answer the question.

THE WITNESS: My answer to your question would be "No."

BY THE COURT:

Q It would be what? A No.

BY MR. SCHIMMEL:

Q. What would you base that on? A Two items
106 appearing under the column headed 150 hours.

BY THE COURT:

Q In which sheet is this? A That is Exhibit 40.

MR. SCHIMMEL: That is Exhibit 40, Your Honor.

THE WITNESS: Under the column under 150 hours, For the normal octyl compound, the rating of the piston per cent filling of the oil ring, and there is a loss in weight of the bearing of 69 miligrams for the capryl compound.

For the normal octy compound, the rating of the piston is D, rather than C, indicating that there were more deposits on the piston, and the ring filling is indicated as 90 per cent for the normal octyl product, which means it was nearly filled, the openings in the rings were nearly filled with sludge, and the bearing had lost 166 miligrams, indicating that there was a higher degree of corrosion.

Q Take 30 hours, for example. Which shows up better there? A That is not significant. I could not tell you. In other words, when two samples are both satisfactory at a given number of hours of operation, there is no distinguishing at that point which is better.

Q You continue the test beyond that, in other words? A That is right.

107 MR. SCHIMMEL: At this point, I am at a loss to understand one thing Mr. Limbach called attention to, and that was the relationship of the early Cook, et al., application with respect to the testimony of this particular witness.

Now, I am not sure whether you are ignoring Cook, et al., because you figure you antedate our reference to the early Cook, et al., application and what is disclosed therein.

MR. LIMBACH: With reference to the early Cook applications?

MR. SCHIMMEL: You made reference to that once. I am not sure to what extent you have put that into the examination.

MR. LIMBACH: It is not in the early application.

MR. SCHIMMEL: The early Cook, et al., application?

MR. LIMBACH: That will stand on its own feet, and it is so clear, I do not think it requires any interpretation.

MR. SCHIMMEL: That is my only question, as to whether it required any cross examination. If you didn't direct examination to it, I won't, as far as that is concerned. We can argue that.

MR. LIMBACH: My feeling is, Mr. Schimmel, that in trying a case of this kind that for the assistance of the Court all of the technical information that we can give the Court for the Court's assistance should be brought into the case.

108 I therefore waive my objection that I might have to your cross examining the witness on the point I didn't touch on direct examination.

You may cross examine the witness as to the early Cook application. I have a copy of it here.

THE COURT: In connection with the testimony of this witness on direct examination, the statement was made with reference to Exhibit 43, containing an abstract of the Cook application, beginning at page 95 of Exhibit 43, that the plaintiff relied on this portion of Exhibit 43 to show the extent of the disclosure of the Cook application, and the statements made in the application which are inconsistent with the statements made in Patent No. 2,344,392, which was assigned to the American Cyanimid Company, and so on.

MR. LIMBACH: My only reason for bringing that in was that I wanted this witness to establish the fact that the assignee of this Cook application was a present competitor in the field with this company.

THE COURT: All right.

MR. LIMBACH: But with regard to the text of the early application, while I didn't examine on that, I have no objection to Mr. Schimmel going into it.

MR. SCHIMMEL: First I will refer to the Cook patent. You have no objection to that, I suppose?

MR. LIMBACH: No.

109 BY MR. SCHIMMEL:

Q I will ask the witness if he will indicate what the invention in that patent is in his opinion? A Claim 1, and I read from it, and it says: A solution of a heavy metal salt of the dicapryl ester of dithiophosphoric acid in lubricating oil.

Q Now, referring to page 101 of Plaintiffs' Exhibit 43, I ask the witness if he will read the first sentence in the last paragraph on that page. A We have discovered that the above and other objects are accomplished when oil-soluble heavy metal salts of dithiophosphoric acid esters are added to crankcase oils in relatively small amounts. Shall I go on?

Q No, that is enough.

Now, on page 108 of that exhibit.

THE COURT: The quotation which the witness just read from page 101, does that relate to the plaintiff claim?

MR. SCHIMMEL: No, that is tying in the Cook patent with this.

THE COURT: That is a description of the claim appearing in the Cook application and not the plaintiffs' patent?

MR. SCHIMMEL: That is right. That throughout run, from the early case to the later case, the heavy metal salt dithiophosphate.

110 BY MR. SCHIMMEL:

Q Now, I call your attention to page 109 of the same exhibit, and will you read the material designated on the second line under the Table 2? A Oil plus .3 per cent barium di-n-octyl dithiophosphate.

Q Now, the particular compound you claim in this case before the Court, what is the difference between that compound and the compound being claimed, the metal? A In this case the metal is barium; in the Asseff application before the Court the metal is zinc.

Q Is zinc a heavy metal? A The answer to that question depends on the person who is defining what he means by heavy metal.

I have seen groups of heavy metals which differ entirely according to the point of view of the user of the group.

Q Do you know what the dictionary says about heavy metals? A A heavy metal presumably would be a metal that is heavy, has a high specific gravity.

MR. SCHIMMEL: I have here a Hackh's Chemical Dictionary which I will ask the Court to take judicial notice of.

BY MR. SCHIMMEL:

Q I show the witness page 631 of that dictionary, showing the periodic system of the elements, and ask you if there does not appear below the middle line, the word heavy metal? A That is correct.

111 Q Can you find zinc listed in the group under heavy metals? A Yes, I do.

Q You find that there? A Yes, sir.

Q On page 632 we have another classification of the elements, and do we find zinc under there listed as a heavy metal? A Yes.

Q As a chemist, would you think you would experience any difficulty following Mr. Cook's suggestion to use a heavy metal in forming his reaction product, to use zinc in place of the one he specifically disclosed? A You are asking me as a chemist whether I think zinc would behave differently from barium?

Q No, I asking you whether there would be any difficulty encountered in substituting a zinc compound for the barium compound?

MR. LIMBACH: I object to that question because the answer that you are trying to get from the witness would seem to indicate that using a zinc compound as an additive, he won't have difficulty making that zinc compound in view of the previous preparation of the barium compound.

The invention in issue is not the method of preparing these materials; the invention in issue is the use 112 of the materials in a lubricant.

We are not claiming, or Asseff is not claiming to have been the first inventor of the method of making these things, but his invention lies in the discovery of the totally unexpected result from the use of a molecule which is characterized as being two principal things, the thiophosphate molecule, and first the presence of zinc, and second, the presence of capryl alcohol residue, giving a totally unexpected thing, so there were two unexpected things. First, there was universal solubility of the zinc compound, and secondly, that is coupled with its totally unexpected effectiveness as an inhibitor as compared with the other metals.

Now, the last question to which Mr. Schimmel is directing his attention has to do with the preparation of materials and so is irrelevant.

THE COURT: Mr. Reporter, will you read the last question?

(The last question was read by the reporter.)

THE COURT: Referring to what?

MR. SCHIMMEL: The preparation of the zinc, and capryl dithiophosphate, which is disclosed in the application, in the Cook patent and the Cook application, and Cook says to use a heavy metal salt.

Zinc being a heavy metal salt, it would seem to me
this person that was a chemist could substitute a
113 zinc for the barium.

I am asking him as a chemist whether that would be so, that is all.

THE COURT: Read the question again.

(The last question was again read by the reporter.)

THE COURT: I think the question is a proper one. I will overrule the objection.

THE WITNESS: As I understand the question now, it is whether there would be any difficulty in the preparation of the compound where zinc were used instead of barium?

BY MR. SCHIMMEL:

Q That is correct. A My answer to that would be "No."

MR. SCHIMMEL: That about finishes my cross examination.

Redirect Examination

BY MR. LIMBACH:

Q On the question of the Cook application and American Cyanimid, was your company on the market with the zinc salt or with a zinc salt inhibitor in the interval between the filing of the original application on—

THE COURT (interposing): That is November 8, 1941, isn't it?

MR. SCHIMMEL: July 11th, Your Honor.

BY MR. LIMBACH:

Q In the interval between the filing of the first
114 application of July 11, 1941, and the filing of the
second Cook application on November 8, 1941?

Will you read the question, now?

(The last question was read by the reporter.)

THE WITNESS: Yes, it was.

BY MR. LIMBACH:

Q It was being sold commercially? A Yes. I think I related that earlier with reference to the time of the filing of the Asseff patent.

Q The earlier Asseff case? A Yes.

If you hand me that, I will tell you the date of that.

Q That is Patent 2,261,047 and that was filed July 28, 1941. A As I recall, that product was offered for sale at about the time that this patent application was filed, so that it was on the market between the time of the filing and the time of the issuance.

MR. LIMBACH: That is all, Your Honor.

Now, in addition to the other exhibits which I have offered, I should like also, since we have referred to them throughout this case—that is all, Doctor.

(Thereupon the witness was excused and retired from the witness stand.)

MR. LIMBACH: Since we have referred to them
115 throughout the examination of the witnesses, I would like to offer these sheets in evidence so they will be in the record as Plaintiffs' Exhibits 48 to whatever the next number comes to. There are eight of them.

May we use the Court's copies? They are originals. It is 48 to 55.

THE COURT: Very well.

MR. LIMBACH: I am sorry, Your Honor, but Mr. Nelson has listed them as 47 to 54.

THE COURT: It should be 47 to 54?

MR. LIMBACH: Yes.

(Thereupon the documents were marked Plaintiffs' Exhibits 47 to 54 for identification, respectively.)

MR. LIMBACH: And they are offered in evidence along with the previous plaintiffs' exhibits numbered from 41 to 46.

THE COURT: Do you have any further testimony?

MR. LIMBACH: That is all, Your Honor.

THE COURT: Very well.

MR. SCHIMMEL: May I present my objection to this evidence now, Your Honor?

THE COURT: You may.

MR. SCHIMMEL: I would like to refer the Court to the chronological history of the Asseff application in the Patent Office, which is Plaintiffs' Exhibit No. 43.
116 Referring specifically to page 35 of that exhibit—

THE COURT (interposing): Mr. Clerk, will you give me the original document, please?

MR. SCHIMMEL: On page 35, there is an office action in which the Cook patent was cited. That action is dated July 22, 1944.

On page 2 of the action, which is page 36 of the exhibit, the first paragraph reads: Claims 1 to 8 are rejected over the Cook patent which discloses the use of zinc salts of the reaction product of capryl alcohol with phosphorus pentasulphide, as improving agent for lubricating composition.

Now, Claims 1 to 8 at that time were all the claims in the plaintiff's application. So on July 22, 1944, he was on notice that someone else was prior to him in the use of that zinc salt as a lubricating additive.

THE COURT: Where do you find the date of July on the exhibit?

MR. SCHIMMEL: It is stamped, U. S. Patent Office, July 22, 1944.

The next office action appears on page 52 of that exhibit, dated March 30, 1945, and on page 52, the last paragraph, that action again called attention to the fact that Cook discloses this specific salt as a lubricating oil improving agent. That action was the final action by the examiner.

117 The applicant appealed the case to the Board of Appeals and on page 55 of the exhibit is the notice of appeal to the Board of Appeals, and Ground No. 5 is a recognition that the examiner at that time unquestion-

ably was relying upon Cook as showing prior use and a prior inventor of that material.

On page 57 is the examiner's statement, mailed on October 16, 1945, in which again the attention of the plaintiff was called to the fact that Cook was prior.

It wasn't until the brief was filed on January 17, 1947, which appears at page 65 of that exhibit, which was two and a half years after the office first called plaintiff's attention to the fact that Cook was prior, that there was any further effort on the part of the plaintiff to produce evidence to show the contrary.

When that evidence was first presented, there was no reason given as to why it had been delayed so long. Presumably it was in the plaintiff's possession all the time, and when that was called to plaintiff's attention, they did file an affidavit as to why it wasn't presented earlier, which was considered insufficient.

We have a situation here, Your Honor—

THE COURT (interposing): Where is the affidavit? Is it in the exhibits?

MR. SCHIMMEL: The affidavit is in the exhibit, beginning on page 139. Mr. Limbach is the affiant in
118 that particular affidavit.

MR. LIMBACH: His Honor is referring to the affidavit under Rule 75.

MR. SCHIMMEL: I thought it was the affidavit of excuse.

THE COURT: It was the affidavit of excuse.

MR. SCHIMMEL: That is on page 139.

THE COURT: Let me read it.

MR. SCHIMMEL: Apparently the only excuse given in that affidavit is—

THE COURT (interposing): Wait until I read the affidavit.

MR. SCHIMMEL: I would like to call Your Honor's attention to the fact that there is nothing in the affidavit that hints at the time that that evidence wasn't previously available, so it seems to me that the conclusion is inescapable that they had it all the time but no effort was made to produce it seasonably.

It is our position that that long interval in which they had the evidence and deliberately withheld it from the Patent Office is sufficient under the rules of the Court of Appeals to exclude this evidence under a 4915 action. I think that is the rule of this jurisdiction.

I would like to read something from the Knutson v. Gallsworthy case that Mr. Limbach brought to your attention yesterday, which was decided September 15, 119 1947, and the Court says this:

"There remains the question of admissibility of certain evidence proffered by Morell in the court below, and denied admission by that court. The rule is well settled that new and additional evidence may be received in a civil action under Section 4915, but it is equally well settled that a party may not successfully offer to the Court evidence withheld from the Patent Office. The answer to the question depends upon whether the latter limitation applies."

It is unquestionably the rule in this jurisdiction that evidence withheld, deliberately withheld from the Patent Office, is not admissible under a 4915 action, and we contend that the facts as shown in this application filed before Your Honor is a deliberate withholding from the Patent Office of the fact that they contend that they were prior to Cook, by waiting two and a half years before hinting at the fact that they were prior to the Cook patent, and I urge that this evidence be excluded under the rule of the Court of Appeals.

THE COURT: Is it your position that this withholding was done in bad faith?

MR. SCHIMMEL: It is deliberate. There is nothing, it seems to me, to indicate in any way that it was other than deliberate.

There is no intimation it was newly discovered
120 evidence, such as might be excusable. It was apparently in their possession all the time, and the Patent Office was led to believe during that two and a half year period that no attempt would be made to contend that they were prior to that reference, and I do not see how it can be held anything other than deliberate. As far as the facts here show, they have no excuse which would indicate otherwise. In fact, no excuse was offered to the office and no excuse is offered here.

It is our position that it was deliberate and the evidence should be excluded.

MR. LIMBACH: First, Your Honor, on the question of time, as to whether two and a half years, as Mr. Schimmel claims, is a long or a short time.

The records of the Patent Office show that there are certain divisions in the Patent Office that are two and a half years behind in reply to amendment. In other words, there are divisions of the Patent Office who have on their desks applications that have been amended two and a half years ago that they haven't yet gotten around to.

In the second place, the affidavit which you read states my position with regard to the manner in which these rejections were developed, and the manner in which I contend that the real issue as to the claims here in issue was not reached until in the final rejection.

121 In other words, the final rejection was the first holding by the Patent Office as to the anticipation of Cook of the claims in the form in which they now appear before Your Honor because they were all amended or rewritten in the previous amendment. Then the delay after the final rejection up to the time of the filing of the antedating affidavit, and there was nothing happened in that interval. There was no action set on the part of the Patent Office, and the position of the Patent Office was not changed in any way.

There is no possibility to go back to the primary inquiry examiner, excepting through and by the consent of the Board of Appeals after the case once goes to the Board of Appeals.

We have come forward with this evidence. We haven't withheld this evidence from the Patent Office. When the Patent Office Board of Appeals, and I consider the Board of Appeals part of the Patent Office, decided the question of the patentability of this invention of this applicant, we came forward with the evidence and said: Here are the facts.

How that can possibly be a deliberate withholding, such as is contemplated by these other cases, I fail to understand.

Now, the language that the Court had in mind in the Gallsworthy case stems from the Koppers case, and in that case there was an interference. It was an
122 inter-party proceeding, where in that inter-party
proceeding in the Patent Office evidence was wilfully withheld in the inter-party proceeding, and then after the proceeding had gone to the District Court, they tried to bring in evidence which they refused to bring in during the Patent Office proceeding. That is the kind of withholding that the Court had in mind in the Gallsworthy case, and there are none of those elements in our situation.

THE COURT: Why didn't you file this material initially? What is the reason why it wasn't filed? You were notified in July, 1941, of the rejection of your claims 1 to 8 upon the ground of the Cook patent.

MR. LIMBACH: The record shows that in response to that office action the claims were changed. At that time all the law offices in the country were very shorthanded, and the man directly in charge of this application may or may not have known at that time of the existence of this evidence. I don't know whether he did or not.

However, there was a full and complete response by either cancelling claims, rewriting them, or amending them, and then in the next office action, which was the final rejection, was the first time that the Cook patent was applied to the claims we have before us.

Then before any further action was taken by the Patent Office, in response to that final rejection, the evidence was brought in. 123

THE COURT: Did you make an effort to file these exhibits with the Patent Office? It is my understanding from the opening statement that you attempted to file the exhibits with the Patent Office and they rejected the file, or rejected the exhibits. Is that correct?

MR. LIMBACH: No, what happened was this. In response to the final rejection, which closes the transaction before the primary examiner, your next step is by way of appeal to the Board of Appeals, and in connection with our presentation of our case to the Board of Appeals, attached to our brief, which we filed 20 days before the hearing before the Board so they would have ample time to look at it and the primary examiner would have ample time to comment on it, that evidence was presented.

Before the Board of Appeals took the case up for consideration, the evidence was in, but they said we should have presented it for consideration by the primary examiner before he issued his final rejection.

Now, as a matter of fact, the only time that the primary examiner ever ruled on the claims in their present form was in the final rejection. That is borne out by the fact that in the amendment proceeding all the claims were rewritten, cancelled, or changed.

THE COURT: What do you say with reference 124 to the statement of counsel that the claims were changed after July, 1941?

MR. SCHIMMEL: I say this, Your Honor: that at no time has plaintiff's counsel ever contended that the Claim 1, for example, wasn't anticipated by Cook, and by this reference. No matter what change he made in it, it is still on Cook. That is the first proposition.

The second proposition is this: Let us assume that before the amended claims were for the first time rejected on Cook, under the final rejection, at least, at that time he knew, which was October, 1945, he knew that the office considered Cook prior to Asseff.

He is incorrect in saying that the office says nothing as to that. The examiner issues, after his appeal is filed, a statement of his reason as to why he thinks the claims are unpatentable, and he issues a statement to the Board of Appeals and the applicant gets a copy of it. So he had two times at which he knew that the examiner considered those claims to be anticipated by Cook.

If you look at the decision of the Board of Appeals in the case, Your Honor, on page 151 of that exhibit, the last paragraph, it states that appellant does not contend that Cook, et al., do not disclose the composition. So there was no real contention made before the Board that Cook didn't have all he claimed for his invention.
125 That was the position he took all along.

He didn't care whether Cook claimed it or not. His position was that merely because some other reference was used, as well as Cook, well, then he didn't have to worry about Cook.

He forgets this: that Cook was used as anticipation against a lot of claims, which means that Cook alone, as far as the office was concerned, was prior, and Salzberg wasn't used. It was used in conjunction with other references. This is an entirely separate and distinct rejection.

If he antedated Cook, he should have eliminated Cook and taken up the case of the rejection on anticipation, leaving only the question of whether there was invention, and whether it was proper for the office to combine these references, which was a different issue.

It is my contention that he has had from October, 1945, when he admits these claims were rejected by the examiner on the final rejection, from October, 1945, to February, 1947, when he presented the evidence, which is an unreasonably long time without some excuse as to why that length of time was necessary under the circumstances of the case.

THE COURT: Do you admit these dates are right, October, 1945 and February, 1947, which he enumerated? The final rejection, October, 1945, and the presentation of these exhibits, in February, 1947?

126 MR. LIMBACH: The final rejection was dated March 30, 1945.

THE COURT: March 30?

MR. SCHIMMEL: Yes, March 30, 1945.

MR. LIMBACH: And then the case went to the Board of Appeals, and our affidavit accompanied the appellant's brief before the Board of Appeals, which was filed about—I haven't that exact date, Your Honor. It was filed about the first of October, 1946.

The hearing date was reset to February 5, 1947, by paper number 13.

You see, on December 15, 1945, the Patent Office recessed the hearing date to 1947, showing that the Patent Office was two years behind. In reaching the case, the delay there, that two-year delay, was due to the crowded condition of the docket of the Board of Appeals of the Patent Office, and they set it over. It was originally set for hearing by a paper dated December 15, 1945. That is final paper number 13.

I don't know what page it is in your exhibit, but on the 14th of December, 1945, they set that case for hearing on the 20th of February, 1947.

THE COURT: In December, 1945, it was set for hearing in February, 1947?

127 MR. LIMBACH: That is right. The Patent Office Board of Appeals docket was crowded and they could not reach it before that time.

THE COURT: When did you file that here, February, 1947?

MR. LIMBACH: February 5, 1947.

THE COURT: When did you file that?

MR. LIMBACH: Along with the brief.

THE COURT: When was your brief filed?

MR. LIMBACH: It went in in January, 1947, presumably. January 17, 1947.

Before the Board of Appeals reached the case, you see.

THE COURT: Do the rules of the Board of Appeals provide that the briefs are to be filed a specified number of days before hearing?

MR. SCHIMMEL: Yes, sir; 20 days.

THE COURT: In other words, this plaintiff was within the time set when he filed the brief on January 17, 1947.

MR. SCHIMMEL: That is correct. He had that time.

THE COURT: Very well.

MR. LIMBACH: I would like to read from the case Your Honor, which I think distinguishes from the Barrett v. Koppers case.

In the case of Globe-Union, Incorporated v. Chicago Telephone Supply Company, 103 Federal (2d) 722, it reads:

> "Counsel goes further and insists that the Court
> 128 in the Barrett case did not restrict the principle announced to situations where the evidence was deliberately withheld or otherwise procurable by the use of due diligence. We disagree with counsel. Every opinion must be read with reference to the facts upon which it is based. The Barrett case stands on facts clearly showing intentional withholding of evidence."

And there it actually was evidence which the witness had been asked to produce and counsel instructed them not to answer.

THE COURT: I am familiar with that case.

MR. LIMBACH: To continue: "To hold with counsel's contention is tantamount to judicial abrogation of

R. S. 4915. Such a contention would make it impossible for a plaintiff to meet the test laid down by the doctrine of Morgan v. Daniels, that is, that the interference judgment can be overcome by testimony which in character and amount carries thorough conviction. We must always be conscious of the distinction between a de novo trial, where the case is tried a second time—" as in a 4915 action—"and the record is made up in the District Court, and a Court's review of findings of an administrative body, where the record is solely that of the administrative body."

That would be in case of an appeal to the Court of Customs and Patent Appeals.

129 It continues: "The instant case falls in the former class."

That was the Globe case. Practically the same thing was stated in the Minnesota Mining case at 155 Fed. (2d) 746.

THE COURT: Is there anything further?

MR. SCHIMMEL: Your Honor, I would just like to call your attention to the administrative problem involved in this situation. The primary examiner is the forum, the expert forum that deals with these inventions. Congress has seen fit to establish the office and provided it with experts to investigate both the novelty and the invention involved in the applications.

The examiner proceeds to make his investigation upon what he considers to be either anticipatory patents, duplications, or those which show what is being claimed is not invention, and he cites them to the applicant, and if the applicant fails to attempt to overcome the effective date of those references, those which can be overcome, he obviously does not look any further.

He figures that here we have something that is admittedly prior, and that applicant has not distinguished from it. It may be that there are other disclosures that could have been used by the examiner.

The applicant in this case delayed the prosentation of his evidence until such time as the case passed out
130 of the jurisdiction of the primary examiner, the expert, and then that evidence is no longer available to either the Board of Appeals or to the Court of Customs and Patent Appeals, or to this Court in the event that a 4915 action is filed.

So that is the situation we have here. The case passed out of the jurisdiction of the primary examiner without any attempt being made to establish that Cook was not prior to Asseff.

The Board of Appeals received that case in that condition, and the experts are no longer available in a situation of this kind when the affidavit is filed. All the evidence presumably is before the Board that the examiner thinks necessary.

From the administrative standpoint, our work is immeasurably burdensome without having situations like this to come up in a case filed as late as it is.

It is true enough that the rules provide, if the Board wishes, it can remand it to the primary examiner, but an applicant should not be permitted to prosecute applications in this fashion because, well, he is not doing what the statute requires him to do, which is to make a bona fide attempt to get his case into final form, so that we can pass on it at the earliest possible moment.

I think the administrative problem is something that the Court should consider.

131 THE COURT: The Court is well appreciative of the administrative problem which confronts the Patent Office in problems of this kind. I think that the situation, both of the applicant and the Patent Office, is analyzed rather well in the single sentence in the case of Schilling, et al., v. Schwitzer-Cummins Company in 79 D. C. Appeals, page 20, appearing at page 21, where the Court points out that one who assumes the difficult burden placed upon a plaintiff in a Section 4915 proceeding does

not aid his cause by giving ground for the impression that he deliberately failed to make a full disclosure in the Patent Office, and that inherent risk of failing to make complete disclosure is such a high one with the Patent Office in dealing with the exigencies of the interest of the applicant to obtain a patent, that it is such that they must make their full disclosures.

In reviewing the case which I have just cited, and its predecessor case which was also cited by counsel yesterday, which is designated Boucher Inventions v. Sola Electric Company, appearing in 76 D. C. Appeals 160, and in a number of other cases cited, cited particularly by plaintiff's counsel, I notice that the words invariably used are suppression or withholding. A number of the decisions refer to deliberately withholding due primarily to the facts of the individual cases necessitating the use of the word deliberately.

132 In the nature of the evidence in question before me, I fully appreciate the delicacy of the words suppression or withholding, and I feel in this case the Court is bound to admit in evidence the exhibits which have been identified by the plaintiff and offered, and I accordingly overrule the Patent Office objection to the admission of these exhibits.

(Thereupon, Plaintiffs' Exhibits 1 to 54, respectively, previously marked for identification, were received in evidence.)

THE COURT: Do you have anything further to offer?

MR. SCHIMMEL: Well, I suppose I should present in evidence the folder that is before the Court, which includes copies of the patents relied upon, the examiner's statement, his reply to the brief and his report, and the decision of the Board of Appeals. That is all I have to offer in evidence.

THE COURT: The clerk will mark the folder as Defendant's Exhibit No. 1.

(Thereupon the document was received in evidence as Defendant's Exhibit No. 1.)

THE COURT: Do you have any other exhibits?

MR. SCHIMMEL: I have no other exhibits.

THE COURT: Do you desire to file briefs in this case?

MR. LIMBACH: Does Your Honor wish that it be submitted on brief? I would prefer it that way.

133 THE COURT: I have no preference. It will be some time before I am caught up in my reading, from the looks of the exhibits.

I have no desire for or against it. It has been my observation that counsel usually desire to file briefs. That is entirely up to counsel. What do you desire to do?

MR. LIMBACH: I would prefer, if it is agreeable to Mr. Schimmel, to submit the case on brief.

THE COURT: Very well. How long do you want in which to submit your brief?

MR. LIMBACH: May I have 30 days?

MR. SCHIMMEL: Do I have time after that?

THE COURT: I will come to you.

MR. SCHIMMEL: That is agreeable to me.

THE COURT: Thirty days for the filing of the plaintiffs' brief. How long after the filing of the plaintiffs' brief do you need in which to make answer?

MR. SCHIMMEL: I think 15 days would be sufficient.

THE COURT: Fifteen days after the filing of the plaintiffs' brief?

MR. SCHIMMEL: Yes, sir.

THE COURT: Very well. This being May 18th, and June 18th being a Saturday, the Patent Office will have 15 days after that. That brings us to about the 1st of July when the Court is in recess. However both
134 sides suggest a period that is acceptable and agreeable to the Court. Plaintiff will have 30 days in

which to file a brief, and the Patent Office will have 15 days after receipt of the plaintiff's brief in which to file an answering brief.

Now, do you want to file a rebuttal brief?

MR. LIMBACH: I will ask the Court's permission at the time, if it seems desirable.

THE COURT: To be filed within 10 days of the Government's brief?

MR. LIMBACH: That is right.

I have one further question and that is with regard to the exhibits.

I have the originals, and they are in the volume from which the witness testified. Your Honor has a book in which there are photostatic copies.

I would like to substitute the photostatic copies that are in Your Honor's volume for the originals of these exhibits.

MR. SCHIMMEL: That is O. K. with me.

THE COURT: They will be substituted.

MR. NELSON: How about Exhibit 36? That is the one to be copied.

MR. LIMBACH: We will see that that is in there.

THE COURT: Very well.

Is there anything further?

MR. LIMBACH: No, sir.

135 THE COURT: Thank you for your very enlightened presentation.

MR. LIMBACH: Thank you, Your Honor.

MR. NELSON: Thank you, Your Honor.

(Thereupon the trial was concluded.)

EXCERPTS FROM PLAINTIFFS' EX. 43

Division of App., No. ...., filed ...., 19.......
L-92 Appeal No. 7873
Number (Series of 1935) Patent No.
1942 Dated
Div. 64 (Ex'r's book) 100-3
Name *Peter A. Asseff*
Assor. to The Lubrizol Corporation, Wickliffe, Ohio, a Corp. of Ohio.
of *Cleveland*
State of *Ohio*
Invention *Lubricating Composition*
Original
Application filed complete *Sept 9,* 1942
Parts of Application Filed Petition, Specification, Oath, First Fee $30 } *Sept 9,* 1942
4 extra claims $4 *Sept 9,* 1942

Examined and passed for Issue ...., 194.. ....Exr. Div.......
Notice of Allowance ...., 194....
By Commissioner.
Final Fee ...., 194....
Attorney *Oberlin, Limbach & Day 909 Leader Bldg Cleveland Ohio*
Associate Attorney ....
No. of Claims Allowed.... Print Claims .... in O. G. Class....
Title as allowed ....

This invention relates as indicated to lubricating compositions and more particularly to compositions which are particularly suited for operation under extremely rigorous conditions such as high temperatures where ordinary lubricants have a tendency to cause the formation of deleterious deposits and the like, which, when such lubricants are used in internal combustion engines such as Diesel engines, result in such deposits causing ring sticking and the like. This invention also relates to improved lubricating compositions characterized by the fact that they are particularly non-corrosive and thus may be used safely with sensitive bearing metals and alloys such as those containing a characterizing amount of silver, cadmium, nickel, lead, copper, tin, iron, etc.

It is a principal object of my invention to provide an improved lubricating composition having the above mentioned desirable properties.

Other objects of my invention will appear as the description proceeds.

To the accomplishment of the foregoing and related ends, said invention then comprises the features hereinafter fully described and particularly pointed out in the claims, the following description setting forth in detail certain illustrative embodiments of the invention, these being indicative, however, of but a few of the various ways in which the principle of the invention may be employed.

Broadly stated this invention comprises the provision of lubricating compositions which contain a characterizing amount of a compound, the molecular structure of which includes a combination of:—

(a) Zinc;
(b) The capryl alcohol radical; and
(c) The di-thio phosphate radical.

More particularly this invention comprises lubricating compositions containing a characterizing amount of the material produced by preparing the zinc salt of the acidic reaction product obtained by reacting phosphorus pentasulphide and capryl alcohol. The compositions of this invention may accordingly also be described more particularly as thus containing a characterizing amount of the zinc salt of dicapryl dithiophosphate, the compound comprising a substantial portion of the reaction product aforesaid.

The present invention is concerned with lubricating compositions in which the above named addition agent is utilized in amounts, hereinafter more particularly stated, in either a base consisting entirely of refined mineral lubricating oil, or such base may advantageously comprise refined mineral lubricating oil to which has also been added other addition agents certain of which, as hereinafter more particularly explained, have a cooperative action with the addition agent particularly identified above.

In the following explanation of the lubricating compositions comprising this invention, it becomes convenient, therefore, to first consider the oil base, second the characterizing addition agent of the present invention with an indication of the mode of its preparation, and third a description of such other addition agents as may be used in conjunction therewith.

(1) *The Oil Base*

The primary component of my improved lubricating composition is a hydrocarbon oil such as mineral lubricating oil. In certain embodiments of my invention it is contemplated to prepare a concentrate which is designed however to be used in preparing a final blend for use, and which blend contains a major proportion of hydrocarbon oil. The concentrate may contain less than a major amount of hydrocarbon oil, or alternatively the hydrocarbon oil may be entirely omitted from the concentrate.

In its principal embodiment, however, my improved lubricant will comprise a major proportion of a hydrocarbon oil such as a mineral lubricating oil or synthetic hydrocarbon lubricating oil. Since my invention may perhaps be defined as the provision of particularly identified materials for use in improving lubricating oils to which they are added, the oil base may be any oil which may be used as a lubricating medium.

A large field of use for lubricants of the character to which my invention relates is in internal combustion engines such as the spark-ignition and Diesel types. It is usually desirable to use for this purpose an oil base which has a high viscosity index, for example, above 55 on the

Dean and Davis scale, and preferably above 75. It is many times advantageous to use an oil base having a viscosity index of 100 or more.

In order to obtain such high V. I. oils from Mid-continent and Coastal crudes, it is essential to subject them to rather drastic solvent extraction methods. Even when such oils are derived from Pennsylvania crudes, there is a distinct advantage in using such drastic refining methods.

Such highly refined oils, and particularly those having a high viscosity index which have been thus drastically refined, while generally improved by such refining, nevertheless, have an increased tendency to be corrosive to certain sensitive metals such as are used for bearings. While the amount of engine deposits formed by such highly refined oils may be reduced by such refining methods, the character of such deposits may become more objectionable. For example, the character of carbon deposits from drastically refined oils is frequently extremely hard.

The viscosity of the oil base used will depend on the type of service for which the lubricant is designed. In general, a compounded lubricant made in accordance with my invention should have a viscosity for a particular use which is the same as that viscosity of a plain mineral lubricating oil used for the same purpose. For example, for most internal combustion engines of the usual automotive type, the viscosity will be within the S.A.E. 10, S.A.E. 20 and S.A.E. 30 ranges; while for heavy-duty and aviation engines higher viscosity ranges are sometimes used, for example, the S.A.E. 40 and S.A.E. 50 ranges.

Throughout the following description of my invention, reference will be made to certain components which should be present in the oil base to provide the improvements to which this invention particularly pertains. It is to be understood that the composition as made up for commercial use may, on occasion, desirably contain such other conventional addition agents as are not incompatible with those to which this invention relates. Thus there may be used in the finished blend, not only the characterizing components of this invention, but also conventional pour point depressors; extreme pressure additions, etc.

(2) The Characterizing Addition Agent

The material, for convenience referred to as addition agent, which characterizes all of the lubricating compositions of this invention is, as indicated, characterized by the fact that its molecular structure comprises in combination:—

(a) Zinc;
(b) The capryl alcohol radical; and
(c) The di-thio phosphate radical.

Such material may be conveniently produced by preparing the zinc salt of the acidic reaction product obtained by reacting capryl alcohol with P_2S_5. This procedure is desirably divided into two stages—first, the preparation of the acidic reaction product, and then the preparation of the zinc salt of the latter.

It should be observed, however, that all three components may be admixed, all at the same time, and the end product produced in a one-step process.

As an indication of the procedure to be followed, Example I given below is a typical laboratory procedure in the preparation of this material, and Example II given below is a typical procedure followed in connection with the preparation of commercial size batches.

Example I

463 grams of capryl alcohol and 158 grams of P_2S_5 were mixed and maintained at a temperature of 100° C. for a period of eight hours, yielding 587 grams of a reaction product having an acid number of 126. To the acidic reaction product then maintained at the reaction temperature of between 90 and 100° C. was added 27.2 grams of zinc oxide, after which the bath temperature was then raised

to from about 150 to about 160° C. in fifteen minutes and held at that temperature for about one hour. The product was then cooled to room temperature and diluted with an equal volume of benzene. The benzene solution was then filtered through paper and the benzene removed by vacuum distillation at absolute pressure of five inches of mercury with a maximum temperature of about 130° C. 205 grams of zinc salt were obtained representing approximately 64% yield. The product upon analysis showed the following:—

Zinc 6.85%
Phosphorus 6.36%
Sulphur 9.44%

Example II

2,975 pounds of capryl alcohol were placed in a reaction vessel at a temperature of about 20° C. and in one hour's time raised to about 85° C., at which temperature the material was held for a period of about three hours. During this latter period 1,030 pounds of finely divided P_2S_5 were slowly added. After the addition of the P_2S_5 was completed, the batch was heated for an additional six hours at 85° C. to 100° C. The last thirty minutes of this latter period was at an absolute pressure of five inches of mercury. The batch was then cooled to 55° C. in one hour, after which 349 pounds of zinc oxide powder were added. The zinc oxide was added over a period of about one hour. The batch was maintained at 55° C. for three hours after all of the zinc oxide had been added, after which the pressure was reduced to five inches of mercury absolute and the batch heated to 100° C., this latter heating step requiring about two hours. This batch produced 3,946 pounds of the zinc salt end product which was dissolved in an equal weight of oil and filtered, using Johns-Manville filter-aid. The analysis of this 50% oil solution was as follows:—

Zinc 3.11%
Phosphorus 3.15%
Sulphur 5.86%

After preparation in the manner indicated above, the oil solution was used as an addition agent in the preparation of following compositions:—

(a) About 67% plain mineral oil, and
  about 43% of the oil solution of the zinc salt of this invention;
(b) About 45% plain mineral oil,
  about 20% of the magnesium phenate derived from lauryl salicylate, and
  about 32% of the oil solution of the zinc salt of this invention; and
(c) About 42% refined mineral oil,
  about 37% calcium salt of mahogany acids, and
  about 21% of the 50% oil solution of the zinc salt of this invention,

and after standing for three months all solutions were clear with no signs of precipitation or separation.

Representative examples of lubricating compositions of the present invention were tested by means of a 2-1/2 H. P. single-cylinder, liquid cooled, four cycle gasoline engine, rated at 1800 R.P.M. The procedure used was as follows:

The engine was loaded by means of a fan type brake which applied a load of 1-1/2 H.P. at 1600 R.P.M. The engine was run for ten minutes at 1200 R.P.M. and then the speed was increased to 1600 R.P.M. The following conditions were maintained constant during the remainder of the test:—

Coolant temperature	345° F.
Oil temperature	225° F.
Air fuel ratio	12.7 : 1

The engine was stopped at 30 hour periods for inspection of piston, piston skirt, piston rings, connecting-rod bearings, etc. The engine was equipped with removable connecting-rod bearings consisting of lead-bronze with a steel backing. These bearings were weighed at the beginning of the test and at each 30 hour inspection period. Before each new test, the engine was thoroughly cleaned and equipped with new piston, piston rings and with new connecting-rod bearings.

In the following Table A will be found a tabulation of several lubricating compositions of the present invention and in following table B will be found a tabulation of the characteristics of the lubricant tested, its comparison with an untreated base oil both as to physical properties and performance in the engine tests.

Table A

Lubricant
No.

1 Untreated S.A.E. 30 solvent-refined Mid-Continent motor oil having V.I. of 85.

2 Same oil as in 1, plus 0.75% of zinc salt of dicapryl dithiophosphate.

3 Same oil as in 1, plus 0.4% of calcium salt of petroleum sulphonic acid.

4 Same composition as in 3, plus 0.25% of zinc salt of dicapryl dithiophosphate.

5 Same composition as in 3, plus 0.50% zinc salt of dicapryl dithiophosphate.

6 Same oil as in 1, plus 1.6% of calcium salt of petroleum sulphonic acid, plus 1.0% of zinc salt of dicapryl dithiophosphate.

7 Same oil as in 1, plus 0.75% of magnesium phenate of lauryl salicylate, plus 1.25% of zinc salt of dicapryl dithiophosphate.

Table B

Engine tests at:—	1	2	3	4	5	6	7
30 Hours:							
Piston rating	D	C	C—	C	A—	A	A
No. of rings stuck	0	0	0	0	0	0	0
Per cent ring-slot filling	0	0	0	0	0	0	0
Bearings loss in mgs.	42	18	173	34	20	9	52
60 Hours:							
Piston rating	E	D	D	C—	B	A	C—
No. of rings stuck	2	0	0	0	0	0	0
Per cent ring-slot filling	40	0	0	5	0	0	0
Bearing loss in mgs.	117	26	341	47	31	....	56
90 Hours:							
Piston rating		D		D	B	A	C—
No. of rings stuck		0		0	0	0	0
Per cent of ring-slot filling		0		10	10	0	5
Bearing loss in mgs.		32		56	41	21	60
120 Hours:							
Piston rating		D		D	C—	B	C—
No. of rings stuck		0		1	0	0	0
Per cent ring-slot filling		0		50	15	0	0
Bearing loss in mgs.		37		68	49	....	57
150 Hours:							
Piston rating		D			C—	B—	C—
No. of rings stuck		0			0	0	0
Per cent ring-slot filling		0			20	5	5
Bearing loss in mgs.		41			57	32	69
180 Hours							
Piston rating						B—	
No. of rings stuck						0	
Per cent ring-slot filling						5	
Bearing loss in mgs.						37	
210 Hours:							
Piston rating						C	
No. of rings stuck						0	
Per cent ring-slot filling						10	
Bearing loss in mgs.						43	
240 Hours:							
Piston rating						C	
No. of rings stuck						0	
Per cent ring-slot filling						10	
Bearing loss in mgs.						49	
Drain analysis:							
Per cent vis. increase	29.0	33.2	43.0	No analysis	36.6	53.7	20.5
Conr. carbon residue	1.19	1.63	1.90		1.11	2.19	0.90
Naphtha insoluble	9.5	1.4	4.0		3.0	3.6	26.2
Acid number	1.28	0.29	1.77		0.40	1.15	0.29

In the following Table C are given the results using treated and ntreated oil secured during a 36-hour Chevrolet engine test, using est procedure as described in "1942 Preprint of the A.S.T.M. 60". Preprint of reports presented at the June 1942 meeting of the .S.T.M.) Appendix IV, pgs. 26 to 40:

Table C

Untreated Solvent-Extracted SAE 20 Motor Oil having VI of about 90

rs. of est	Con. Carb. Residue	Acid No.	% Ash	Naph. Ins.	$CHCl_3$ Sol.	Vis. in SUS at 100° F.	% Vis. Inc.	Milligrams Loss in Wt. of Bearings	
								Rod 3	Rod 4
0	.02	.08	0	....	....	335	....		
8	.29	.83	.04	26.8	6.8	369	10.2	189	200
16	.82	1.65	.13	55.8	23.2	430	28.5	Eng. badly	
24	1.23	2.40	.11	66.4	33.4	499	49.4	Varnished	
36	2.09	3.13	.16	162.8	93.0	626	87.0		
Same Oil Treated With .5% of the Zinc Salt of Di-capryl Di-thiophosphate									
0	.14	.45	.09	....	....	323	....	70	109
8	.38	.53	.09	20.4	5.0	322	....	Eng. fairly	
16	.71	1.21	.15	36.7	18.0	337	4.3	Clean	
24	.99	1.83	.21	59.1	22.2	423	31.1		
36	1.03	2.14	.17	71.5	39.3	429	33.0		

(3) *Other Addition Agents Which May Be Used in Combination with the Characterizing Addition Agent.*

It has been found that while the characterizing addition agent above defined possesses the property of inhibiting, to some extent, the formation of harmful deposits in internal combustion engines in addition to that of a corrosion inhibitor in that it reduces the amount of deleterious deposits which occur when engines such as Diesel engines lubricated thereby are operated at high temperatures or for a substantial length of time, nevertheless, such characterizing addition agent also has a cooperative effect when used in the same lubricant with other materials which are effective detergents.

By the term detergent, I mean to include those materials which when added to a hydrocarbon lubricating oil will substantially reduce the amount of deleterious deposits formed in an internal combustion engine when the oil is used as a crank case lubricant.

Examples of such materials are, in general, stable oil-soluble metal-containing organic compounds, and more particularly such compounds which contain the -XM radical where M is a metal, and X is an element of the class consisting of oxygen, sulphur, and selenium. It will be noted that these three elements are members of Group VI of the Periodic Table, oxygen being the first member of this group, sulphur and selenium being the next two members in the right-hand column of such group. In other words, these are the first three non-metallic elements of Group VI.

The -XM radical in such detergents may be in the form of the —C-X-M radical, such as in the metallic alcoholates, phenates, carboxylates, and the corresponding thio compounds. Too it may be in the form of the Y-X-M radical, where Y is an acid forming element, such as in the salt-esters of phosphoric, phosphorous, sulphuric and sulphurous acids and the corresponding thio acids; and in the salts of phosphonic, phosphinic, sulphonic, and sulphinic acids, and the corresponding thio acids. Additional examples of such detergents are the corresponding compounds derived by substituting arsenic for phosphorous, and selenium for sulphur.

Reference to various classes of such detergents, as well as a list of specific examples which will be found advantageous for use, and which will cooperate with the characterizing addition agent previously defined, is found in my patent No. 2,261,047, page 4, first column, line 6, to page 4, second column, line 9.

For a further enumeration of classes, sub-classes, and specific examples of detergents which may thus be used in combination with the characterizing addition agent of this invention, reference may be had to the copending application of Carl F. Prutton, Serial No. 416,753 filed Oct.

27, 1941, and particularly to that portion of the specification captioned "The Detergents", beginning at the bottom of page 5 of the specification of that application. I, therefore, intend to include in this disclosure the teaching that the characterizing addition agent of the present invention may be advantageously used in combination with any of the detergents thus generically, sub-generically, or specifically defined in said Prutton application. Since reference may be had to said Prutton application for such identification of the detergents, it is believed unnecessary to reproduce herein that portion of the Prutton specification referred to.

As previously indicated, the oil base may also contain amounts of other addition agents effective for particular purposes such as pour point depressors, extreme pressure addition agents, and the like. When used under conditions of extreme stress, where high unit pressures are encountered, and where it is desired to reduce to a minimum the wearing or scuffing of the relatively moving metal parts of the engine for example, then the composition of my invention may desirably also contain a substantial amount, on the order of .1% to about 10% of a halogen bearing organic compound. Such halogen bearing organic compound should be stable, oil-soluble, and have a vapor pressure less than atmospheric at a temperature of 140° C. and preferably at a temperature of 170° C. When the lubricant is intended for use as a crankcase lubricant, the halogen compound should desirably be an organic ring compound and preferably of the type in which the halogen is attached to a benzenoid ring structure.

Examples of halogen-bearing compounds, generally, and of halogenated organic ring compounds of the type referred to above, are found in Prutton Patent No. 2,276,341, page 2, first column, line 19, to page 3, second column, line 43.

It is frequently advantageous to use combinations of different classes of halogen compounds, instead of a single halogen compound. For example, in motor oils it has been found useful to use a combination of a halogenated oxygen-free compound, together with a halogenated oxygen-containing compound. Ortho-dichlorbenzene and chlorinated diphenyl ether are examples of these two classes of compounds. The following composition is an example of a crankcase lubricant containing such halogen-bearing constituents:—

Zinc salt of this invention	0.75%
o-dichlor benzene	0.5%
hexachlor diphenyl ether	0.5%
S.A.E. 20 solvent-extracted Mid-Continent motor oil	98.25%

The ultimate composition, when designed for use in environments where a substantial amount of moisture is present, as in turbines, and where the conditions encountered strongly promote rusting especially of iron and steel surfaces, may desirably also contain an additional corrosion inhibitor of another type.

Among the several corrosion inhibitors thus available for this particular use, the following will be found of particular utility:—

p-amino phenol
B-naphthol
α*-naphthyl amine*
catechol
phenyl phenylene di-amine
phenyl dicyclohexyl amine
phenyl α-naphthyl-amine
phenyl thiomorpholine

Since the lubricating compositions of the present invention are designed primarily for use as lubricants in Diesel engines, I shall list below a number of specific examples of finished compositions designed particularly for this specific use. In the following specific examples, I shall, for convenience, identify the characterizing addition agent of the present invention as "zinc dicapryl dithiophosphate". However, it is to be understood that this term is used in a generic sense in that it, when thus used, indicates not only such compound, irrespective of the manner of its formation, but also includes the material produced by preparing in the manner indicated above the zinc salt of the reaction product of capryl alcohol and phosphorous pentasulphide.

Example 1	% By Weight
Mineral Oil S.A.E. 20; 95 V.I.	99.25
Zinc dicapryl dithiophosphate	0.75
Example 2	
Mineral Oil S.A.E. 20; 105 V.I.	99.00
Example 3	
Mineral Oil S.A.E. 30; 35 V.I.	97.4
Zinc dicapryl dithiophosphate	1.0
Ca salt of "mahogany acids"	1.6
Example 4	
Mineral Oil S.A.E. 30; 95 V.I.	98.0
Zinc dicapryl dithiophosphate	0.5
Ca phenate derived from di-(tert-amyl-hydroxy-phenyl)sulphide	0.5
Ca salt of "mahogany acids"	1.0
Example 5	
Mineral Oil S.A.E. 30; 5 V.I.	98.5
Zinc dicapryl dithiophosphate	0.5
Ca phenate derived from di-(tert-amyl-hydroxy-phenyl-sulphide	1.0

From the foregoing specific examples it will be observed that the characterizing addition agent of the present invention may be used in amounts within the range of from about 0.1% to about 4% based on the weight of the total composition. For most uses the amount of the characterizing addition agent required for optimum results (including considerations of cost, etc.) will lie within the range of from about .5% to about 2.5%, based on the weight of the total composition. It will likewise be observed that since one of the principal uses for which the invention is designed is the lubrication of Diesel engines, the base oil may have a viscosity index extending over a wide range as for example from about 0 to about 120 (Dean & Davis), depending upon the requirements of the particular types of Diesel engines in which the lubricant will be used.

For certain types of engines, best results will be secured when the viscosity index lies within the range of from about 0 to about 50. For other types of engines requiring a high viscosity index oil, the base oil may desirably have a viscosity index of at least 50, and for such uses, preferably above 85.

Other modes of applying the principle of my invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims or the equivalent of such be employed.

I, therefore, particularly point out and distinctly claim as my invention:—

1. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorous pentasulphide.

2. A lubricating composition containing a characterizing amount of the zinc salt of dicapryl dithiophosphate.

3. A lubricating composition comprising a major proportion of mineral lubricating oil and a minor proportion of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorous pentasulphide.

4. A lubricating composition comprising a major proportion of mineral lubricating oil and from about 0.1% to about 4% of the zinc salt of dicapryl dithiophosphate.

5. A lubricating composition for Diesel engines capable of inhibiting ring sticking and corrosion of the bearing

surfaces of the engine with which the same comes in contact, especially when such bearing surfaces comprise a characterizing amount of a metal of the class consisting of nickel, copper, silver, cadmium, lead and tin, comprising a major proportion of mineral lubricating oil and a minor amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorous pentasulphide.

6. A lubricating composition for Diesel engines capable of inhibiting ring sticking and corrosion of the bearing surfaces of the engine with which the same comes in contact, especially when such bearing surfaces comprise a characterizing amount of a metal of the class consisting of nickel, copper, silver, cadmium, lead and tin, comprising a major proportion of mineral lubricating oil and from about 0.1% to about 4%, based on the weight of the total composition, of the zinc salt of dicapryl dithiophosphate.

7. An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide.

8. An addition agent capable of improving the properties of a lubricating oil affecting its use in the lubrication of Diesel engines, comprising a concentrated solution in mineral oil of the zinc salt of dicapryl dithiophosphate.

9. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a separate detergent.

10. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a metal-containing detergent.

11. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a calcium salt of a petroleum sulphonic acid.

12. An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a separate detergent.

13. An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a metal-containing detergent.

14. An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a calcium salt of a petroleum sulphonic acid.

15. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and from about .1% to about 20% of a stable oil soluble halogen bearing organic compound having a vapor pressure less than atmospheric at 140° C.

16. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a separate oil-soluble organic corrosion inhibitor.

17. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide and a separate detergent, and from about .1% to about 20% of a stable oil-soluble halogen-bearing organic compound having a vapor pressure less than atmospheric at 140° C.

18. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, a separate detergent, and a separate oil-soluble organic corrosion inhibitor.

19. An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, a separate detergent, and from about .1% to about 20% of a stable oil-soluble halogen-bearing organic compound having a vapor pressure less than atmospheric at 140° C.

20. An addition agent capable of inhibiting ring sticking and corrosion for lubricating oils used in the lubrication of Diesel engines comprising a concentrated solution in mineral oil of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, a separate detergent, and an oil-soluble organic corrosion inhibitor.

21. A lubricating composition containing a characterizing amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, a separate detergent, from about .1% to about 20% of a stable oil-soluble halogen bearing organic compound having a vapor pressure less than atmospheric at 140° C., and an oil-soluble organic corrosion inhibitor.

22. A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and an oil-soluble metallic phenate.

23. A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and an oil-soluble metallic phenate derived from an ester of salicylic acid.

24. A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and the magnesium phenate derived from lauryl salicylate.

Signed by me this 5 day of September, 1942.

/s/ Peter A. Asseff

OATH

State of Ohio
County of Cuyahoga ss.

PETER A. ASSEFF being duly sworn, deposes and says that he is the applicant herein, a citizen of the United States and a resident of Cleveland, Ohio; that he does verily believe himself to be the original, first and sole inventor of the Improvement in

"LUBRICATING COMPOSITION"

described and claimed in the foregoing specification, signed by him; that he does not know and does not believe that the same was ever known or used before his invention or discovery thereof, or patented or described in any printed publication in this or any foreign country before his invention or discovery thereof, or more than one year prior to his application in this country, or in public use or on sale for more than one year prior to his application in this country; that his invention has not been patented in any country foreign to the United States on an application filed by him, or his legal representatives or assigns more than twelve months prior to his application in this country; and that no application for patent on same has been filed by him, or his legal representatives or assigns, in any foreign country.

/s/ Peter A. Asseff

Sworn to and subscribed before me, this 5th day of September, 1942.

/s/ J. R. Winch
Notary Public

My Commission Expires Dec. 5, 1943.
(SEAL)

* * * *

This application has been examined and the following references made of record:

Salzberg	2,063,629	Dec. 8, 1936	252-48
Asseff	2,261,047	Oct. 28, 1941	252-48xr
Wilson	2,280,419	Apr. 21, 1942	252-33
McNab	2,289,795	July 14, 1942	252-48
Frey	2,296,433	Sept. 22, 1942	252-48xr

Claims 1 to 8 are rejected over the patent to McNab. This patent discloses as addition agent in lubricating oil where extreme pressures are encountered calcium salts of dicapryl dithiophosphate. Since calcium and zinc are in the same periodic group, this compound is deemed to be the full equivalent of applicant's compound.

Claims 1 to 8 are further rejected over the patent to Salzberg. This patent discloses the reaction of straight chain high molecular weight alcohols with phosphorus pentasulfide which are then reacted with zinc or calcium to form the salts. Calcium and zinc are considered as equivalents in this patent.

Claims 9 to 24 are rejected over the patent to McNab in view of the patent to Asseff. The patent to McNab discloses the use of metal salts of dicapryl dithiophosphate as addition agents to lubricating oils. The patent to

22. A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and an oil-soluble metallic phenate.

23. A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and an oil-soluble metallic phenate derived from an ester of salicylic acid.

24. A lubricating composition containing characterizing amounts of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide, and the magnesium phenate derived from lauryl salicylate.

Signed by me this 5 day of September, 1942.

/s/ Peter A. Asseff

OATH

State of Ohio
County of Cuyahoga ss.

PETER A. ASSEFF being duly sworn, deposes and says that he is the applicant herein, a citizen of the United States and a resident of Cleveland, Ohio; that he does verily believe himself to be the original, first and sole inventor of the Improvement in

"LUBRICATING COMPOSITION"

described and claimed in the foregoing specification, signed by him; that he does not know and does not believe that the same was ever known or used before his invention or discovery thereof, or patented or described in any printed publication in this or any foreign country before his invention or discovery thereof, or more than one year prior to his application in this country, or in public use or on sale for more than one year prior to his application in this country; that his invention has not been patented in any country foreign to the United States on an application filed by him, or his legal representatives or assigns more than twelve months prior to his application in this country; and that no application for patent on same has been filed by him, or his legal representatives or assigns, in any foreign country.

/s/ Peter A. Asseff

Sworn to and subscribed before me, this 5th day of September, 1942.

/s/ J. R. Winch
Notary Public

My Commission Expires Dec. 5, 1943.
(SEAL)

* * * *

This application has been examined and the following references made of record:

Salzberg	2,063,629	Dec. 8, 1936	252-48
Asseff	2,261,047	Oct. 28, 1941	252-48xr
Wilson	2,280,419	Apr. 21, 1942	252-33
McNab	2,289,795	July 14, 1942	252-48
Frey	2,296,433	Sept. 22, 1942	252-48xr

Claims 1 to 8 are rejected over the patent to McNab. This patent discloses as addition agent in lubricating oil where extreme pressures are encountered calcium salts of dicapryl dithiophosphate. Since calcium and zinc are in the same periodic group, this compound is deemed to be the full equivalent of applicant's compound.

Claims 1 to 8 are further rejected over the patent to Salzberg. This patent discloses the reaction of straight chain high molecular weight alcohols with phosphorus pentasulfide which are then reacted with zinc or calcium to form the salts. Calcium and zinc are considered as equivalents in this patent.

Claims 9 to 24 are rejected over the patent to McNab in view of the patent to Asseff. The patent to McNab discloses the use of metal salts of dicapryl dithiophosphate as addition agents to lubricating oils. The patent to

Asseff discloses that calcium salts of mahogany acids are well known detergent agents and magnesium phenate of lauryl salicylate is used as a corrosive inhibiting agent. No invention resides in adding these compounds to a McNab composition and obtaining expected results.

Claims 9 to 24 are further rejected over the patent to McNab in view of the patent to Wilson. The patent to Wilson discloses the use of calcium salts of sulfonic acid as detergent agent and metal phenates as anti-corrosive agents. These claims are, therefore, rejected for reasons stated above.

Claims 9, 10, 12, 13, 15, 16, 17, 18, 19, 20, 21, and 22 are rejected as being too broad and indefinite as to the detergent, corrosion inhibitor, and halogen compound used. Applicant has found certain compounds useful for his purpose and should limit himself to them and not claim detergents, corrosion inhibitors, and halogen compounds generally.

All claims are rejected.

/s/ P. M. Nash
Examiner

CGP-pef

* * * *

Hon. Commissioner of Patents,
Sir:

In response to Office action of Jan. 20, 1943, applicant hereby replies as follows:—

The references cited and the Examiner's application of the same to the claims herein has been carefully considered, and since it is believed that the references are not anticipatory of the applicant's invention as defined by the claims, reconsideration of the application is respectfully solicited.

The principal reference relied upon by the Examiner is the patent to McNab No. 2,289,795. This is a fairly recent patent. It represents, therefore, very recent information by one who can certainly be considered as "skilled in the art" since he is employed in what is perhaps the largest lubricant research laboratory in the world. It would seem appropriate, therefore, to turn to the specification of the McNab patent for information on the very points on which the Examiner has rejected the present case.

McNab, after referring to the fact that only in recent years have certain polyvalent metal soaps been used in crankcase lubricants such as Diesel engines, and after making further reference to the fact that the function of such soaps is quite complex, states:—

"The various soaps have peculiarities in behavior *depending on the particular metals* and to some degree the acid radicals with which they are combined." (Page 1, first column, line 17).

After referring to different degrees of solubility of the various soaps, McNab continues:—

"Still others are capable of giving clean engines, but do not appear to be able to reduce engine wear or perhaps even cause excessive engine wear." (Page 1, first column, line 23).

McNab here states the new well-established fact that the function of giving a clean engine is not the only function which must be taken into account in the selection of a particular soap for commercial use. McNab says further:—

"Thus the various soaps, while their functions may in some cases overlap, are not equivalents in some certain respects." (Page 1, first column, line 25).

At a later point in the same column, at line 39, McNab reiterates:—

"The results produced by these various soaps as stated above overlap to some extent, but nevertheless they do not appear to be full equivalents."

As pointed out in the first quotation from the McNab specification, perhaps one of the most critical factors which determines whether a particular soap will function satisfactorily is "the particular metal" present, and also "the acid radicals" with which the metal is combined.

In order to provide a lubricant which was not only a satisfactory detergent but at the same time non-corrosive, McNab proposed the use of combinations of very carefully selected metals. That McNab was most careful in the selection of a metal is evidenced by the fact that in the primary statement of his invention found in the first column, beginning with line 29, he groups various metals into three classes; class 1 comprising calcium and magnesium; class 2 comprising aluminum; and class 3 comprising nickel. He states that his invention is concerned with the combined use of two metals respectively selected from different classes as above provided. At no place whatever in the specification does McNab ever mention zinc. He did not mention zinc because he was of the opinion that zinc would not work, or, at most, he did not mention zinc because he was unable to theorize whether or not zinc might work since from the record as given in his specification he had never tried zinc.

The Examiner states that the McNab patent discloses the use of calcium salts of dicapryl dithiophosphate. This is not a strictly accurate statement. The Examiner presumably had reference to page 3, column 2, line 70, where calcium dioctyl dithiophosphate is given as a specific example. Capryl alcohol is an octyl alcohol, but there are octyl alcohols which are not capryl. Capryl alcohol is a secondary alcohol whereas the normal octyl alcohol such as is referred to by McNab is a primary alcohol and the respective derivatives are quite different in character and behavior.

Thus, the McNab patent fails as a reference for two principal reasons:—

(1) While specifically recognizing that the selection of the metal is critical, McNab does not mention zinc; and

(2) McNab does not specify the capryl compound.

One of the other references largely relied upon by the Examiner is the patent to Salzberg No. 2,063,629. While that patent has in it a few loose references to metal salts of thiophosphates, it has long since been recognized by the Patent Office that Salzberg's specification does not teach the invention of using particular sub-generic groups and specific examples, especially in Diesel engine lubricants.

While calcium and zinc may both be mentioned by Salzberg, it would, nevertheless, be noted that one of the principal purposes for which Salzberg's products was to be used was as flotation agents, and further that his invention is primarily concerned with the esters since that is all that he claims, and reference to the salts is entirely incidental. Perhaps calcium and zinc are equivalent in flotation agents, but it has been clearly established by workers such as McNab that the various metals are not equivalents and the present applicant has found, as demonstrated by the data made a part of the specification, that calcium is not equivalent to zinc.

Actually Salzberg fails completely as a reference since his disclosure is limited to alcohols having at least 10 carbon atoms whereas capryl alcohol contains 8 carbon atoms.

The remaining rejections, where the Examiner combines McNab with other patents such as Asseff and Wilson, fall with the failure of the McNab patent as a reference. The applicant has discovered that entirely unexpectedly improved results are secured by the selection of his particular addition agent, the use of which in lubricating compositions is novel broadly. He has found that this novel agent of unusual effectiveness is cooperative with certain other materials. A rejection which is based on a combination of references, when such references are cited merely for the purpose of showing separately the several components of the combination, certainly falls when, as in the present case, it is shown that one of the applicant's components is per se new.

On the last rejection, namely, that of undue breadth, this is believed improper since the applicant has found that his improved addition agent is useful and cooperative with the broadclass of other additives included in the claims.

It is, therefore, believed that all of the claims now standing in this application are clearly allowable, and favorable reconsideration is accordingly respectfully requested.

Respectfully submitted,
/s/ Oberlin, Limbach & Day
Attorneys

* * * *

Hon. Commissioner of Patents,
Sir:

In supplemental response to the Office Action of January 20, 1943 we hereby reply as follows:—

It is believed that it may be helpful to summarize the preceding amendment, pointing out the various reasons why the prior art is not anticipatory of the claims herein.

1. Whenever the term "Octyl" is used, as in "Octyl alcohol", the normal primary alcohol or corresponding derivative is intended.

2. Capryl alcohol, while an eight carbon atom alcohol, is, however, a secondary alcohol, and quite different in character and behavior from the normal primary compound.

3. McNab mentions the "Octyl" derivative but not the "capryl".

4. McNab shows that the action of the various metals is specific and not equivalent.

5. McNab does not name zinc.

6. Salzberg while incidentally including zinc in a large list of metals is concerned with derivatives of alcohols containing more than 10 carbon atoms. Salzberg says that those having less than 10 are inoperative because derivatives of them are not oil soluble.

7. Applicant has provided a *specific* material which brings together in a single molecule:—

(a) The specific metal, zinc.

(b) The capryl radicle, derived from a secondary alcohol.

(c) The di-thiophosphate radicle.

8. There is no suggestion whatsoever in any of the references which would lead one to even *try* this specific combination. On the contrary:

(a) Salzberg would say it wouldn't work because not oil soluble.

(b) McNab would say it wouldn't work because the specific metal *zinc* is not recognized by him as operative.

(c) The capryl radicle has never been suggested by anyone.

Respectfully submitted,
/s/ Oberlin, Limbach & Day
Attorneys

* * * *

Potter	2,246,415	June 17, 1941	252-46
Rutherford	2,252,985	Aug. 19, 1941	252-39

Claims 1-8 are rejected over the patent to Potter. This patent discloses as an effective corrosion inhibitor in lubricating oils zinc salt of octyl thiophosphate. Since applicant has failed to show wherein improved and novel results are obtained by using a capryl radical instead of the octyl radical which also contains 8 carbon atoms in the aliphatic chain, the two are held to be equivalent and no invention seems to reside in substituting the capryl alcohol for the octyl alcohol and obtaining obvious and expected results.

Claims 9-24 are rejected over the patent to Potter in view of the Asseff patent for reasons stated in last Office action when the McNab patent was used instead of the Potter patent.

Claims 9-24 are further rejected over the patent to Potter in view of the Wilson patent cited in last Office action.

Claims 9, 10, 12, 13 and 15-22 are rejected on the ground of indefiniteness and breadth. This rejection is regarded as being sound and is repeated.

No claims are allowed.

/s/ P. M. Nash
Examiner

* * * *

Hon. Commissioner of Patents.
Sir:

In response to Office action dated Oct. 4, 1943, we hereby amend as follows:

Claims 9, 10, 12 and 13, last line of each, and claim 17, line 4, claim 18, line 4, claim 19, line 6, claim 20, line 6, and claim 21, line 4, after each occurrence of "detergent" insert —of the class consisting of oil-soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur—.

Remarks

The additional references which have been cited have been carefully considered. Certain of the claims have been amended to somewhat more clearly define the applicant's invention and reconsideration of the application is respectfully solicited.

In the first principal paragraph of the Office action, claims 1 to 8 were rejected as unpatentable over Potter. This rejection is not understood and the Examiner is respectfully requested to explain such rejection in greater detail since the Patent Office has recognized that the capryl alcohol is patentably distinct over octyl alcohols generally as evidenced by the allowance of patent No. 2,344,392 to Cook et al on March 14, 1944. In view of the fact that the Patent Office has thus recognized the patentable distinction between capryl and octyl alcohols generally, it is believed that the Potter patent is removed as a reference without further consideration.

In the second paragraph in said Office action, claims 9 to 24 were rejected as unpatentable over Potter in view of Asseff citing the applicant's own prior patent. In view of the fact that the Potter patent is not a reference for the reasons above explained and already recognized by the Patent Office, it becomes evident that such patent may not be combined with the applicant's own prior patent in seeking to spell out a rejection of the claims.

In the third principal paragraph of the last Office action, claims 9 to 24 were rejected as unpatentable over Potter in view of Wilson. The same comments made above with respect to the combination of Potter and Asseff applies to this combination of Potter and Wilson, i. e. since the Potter patent is not a reference it is believed improper to combine it with the Wilson patent in spelling out an anticipation of these claims.

In the pre-ultimate paragraph of the Office action, claims 9, 10, 12, 13 and 15 to 22 were rejected on the ground of indefiniteness. It is believed that this rejection has been overcome by the amendments made by the changes in such claims by the present amendment in which the detergent is specifically defined. With regard to the claims which call for the combination of the named primary addition agent and a separate oil-soluble organic corrosion inhibitor or halogen-bearing organic compound, reference may be had to the specification where such terminology in its generic sense is used and supporting data is given on pages 8a, 8b and 8c showing a cooperation between the named addition agents. It is a well established rule of law that a patent specification may be its own lexicon and accordingly since the present specification uses the precise terminology employed by the claims and clearly defines what is meant to be included by the wording of the claims, it is believed that the claims are neither indefinite nor of undue breadth especially since there is no art which shows the claim combinations. Under the circumstances therefore, it is believed that the claims are not anticipated and favorable reconsideration of the application is respectfully solicited.

Respectfully,
/s/ Oberlin, Limbach & Day
Attorneys.

* * * *

Responsive to amendment filed April 3, 1944.
Added art:

Salzberg	2,063,629	Dec. 8, 1936	252-48
Cook	2,344,392	Mar. 14, 1944	252-39

Claims 1, 2, 9, 10, 11, 16, 17, 18, 21, 22, 23 and 24 are rejected as being vague and indefinite as to the use of the expression "characterizing amount". The expression fails to indicate what properties applicant's oil additive imparts to a lubricant when admixed with it or just exactly what amount one might call a "characterizing amount".

Claims 16, 18, 20 and 21 are rejected as being too broad and indefinite as to the use of the term "corrosion inhibitor". In spite of applicant's broad disclosure no basis can be found in the specification to include any and all corrosion inhibitors for lubricating compositions.

Claims 9, 10, 12, 13, 17, 18, 19, 20, 23 and 24 are rejected on the ground of insufficient disclosure as to the detergents claimed. Applicant refers to a copending application of Carl F. Prutton for detergent products which could be used in combination with applicant's addition agent of the present alleged invention. Each application must be complete in itself without having to refer to another application for material applicable to the application under consideration. See Ex parte Kellogg, 1906 C. D. 89.

Claims 1-8 are rejected over the Cook patent which discloses the use of zinc salts of the reaction product of capryl alcohol with phosphorus pentasulfide, as improving agent for lubricating composition.

Claims 1-8 are further rejected over the Salzberg patent which discloses the use of zinc salts of reaction products obtained by reacting a secondary alcohol with phosphoruc pentasulfide, as additives for lubricants.

Claims 9-24 are rejected over the Cook and Salzberg patents in view of the patents to Wilson and McNab cited in previous office action. The Cook and Salzberg patents have been discussed in the previous paragraphs. The Wilson and McNab patents disclose the use of corrosion inhibitors in combination with detergent agents similar to those disclosed by applicant. In view of this teaching no invention seems to reside to add well known detergent to a Cook composition and obtaining obvious and expected results.

No claims are allowed.

/s/ P. M. Nash
Examiner

GGP/n

* * * *

Hon. Commissioner of Patents,
Sir:

In response to the Office action dated July 22, 1944, applicant hereby amends as follows:—

Claim 1, line 2, cancel "preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide" and insert in lieu thereof—the simultaneous admixture of capryl alcohol, phosphorus pentasulphide. and zinc oxide—.

Claim 5, line 6, after "of" insert —highly refined solvent extracted—. Claim 5, line 7, after "oil" insert —having a viscosity index of the Dean & Davis Scale of over 75—.

Cancel claims 2, 3, 4, 6, 7 and 8 without prejudice to the applicant's right to include the subject matter thereof in a divisional or a continuation application.

Page 10, cancel lines 9 to 23 inclusive and insert in lieu thereof—

Compounds of the latter above mentioned types may have the general formula: $R_{(n-2a-b)}X_aY(X'M)_b$

Where:

R is a radicle selected from the class consisting of R'- and R'-X''-, in which

R' is an organic radicle,

X, X' and X'' are the same or different elements of the class consisting of oxygen, sulphur and selenium; -X when present being connected to Y by a double bond,

M is a metal,
Y is an acid-forming element,
n is the valence of Y,
a is an integer which may be zero,
b is an integer which is at least one
(n-2a-b) is at least one.

The organic radicles which, for example, are included within the scope of R′ in the above formula are such as the following:—

Table No. 1

I. Hydrocarbon radicles
  A) Alkyl radicles
    Amyl (n, iso, sec. and tertiary)
    Lauryl
    Cetyl
    Carnaubyl
    Ceryl
    Montanyl
    Mellisyl
    Myricyl
    Lacceryl
    Psyllicyl
  B) Aryl radicles
    Phenyl
    Xenyl
    Naphthyl (alpha- and beta-)
    Anthracyl
    Phenanthryl
  C) Cyclo-alkyl radicles
    Cyclopentyl
    Cyclohexyl
    Cycloheptyl
    Cyclohexyl-cyclohexyl
  D) Alkylated aryl radicles
    Radicles formed by the alkylation of aryl radicles, such as those listed above (B), with any of

the alkyl radicles, e. g. those listed above (A), or with any of the following:—

Methyl
Ethyl
Propyl (n-, and iso-)
Butyl (n-, iso- and sec-)

Specific examples of alkylated aryl radicles are as follows:—

Cresyl
Xylyl
Trimethyl-phenyl
Ethyl-phenyl
Tetra-ethylphenyl
t-Amyl-phenyl
Lauryl-phenyl
Cetyl-phenyl
Ceryl-phenyl
Methyl-naphthyl
Iso-propyl-naphthyl
t-Amyl-naphthyl
Lauryl-naphthyl
Cetyl-naphthyl
Iso-propyl-xenyl
t-Amyl-xenyl
Lauryl-xenyl

E) Alkylated Cyclo-alkyl radicles

Radicles formed by the alkylation of cycloalkyl radicles, such as those listed above (C), with any of the alkyl radicles, such as those listed above. Specific examples are:—

Methyl-cyclohexyl
Dimethyl cyclohexyl
t-Amyl cyclohexyl
Lauryl cyclohexyl
Cetyl cyclohexyl

F) Cyclo-alkylated aryl radicles, e. g.
  - Cyclohexyl-phenyl
  - Cyclohexyl-xenyl
  - Cyclohexyl-naphthyl

G) Arylated-alkyl radicles, e. g.
  - Benzyl
  - Phenyl-ethyl
  - Phenyl-amyl
  - Phenyl-lauryl
  - Phenyl-cetyl
  - Xenyl-methyl
  - Xenyl-ethyl
  - Naphthyl-methyl
  - Naphthyl-ethyl

H) Cyclo-alkylated alkyl radicles, e. g.
  - Naphthenyl
  - Hexahydrobenzyl
  - Cyclohexyl-ethyl
  - Cyclohexyl-amyl

I) Arylated cyclo-alkyl radicles, e. g.
  - Phenyl-cyclohexyl
  - Xenyl-cyclohexyl
  - Naphthyl-cyclohexyl

J) Mixed radicles, e. g.
  - Methylcyclohexyl-phenyl
  - Amylcyclohexyl-phenyl
  - Cyclohexyl-cresyl
  - Cyclohexyl-xylyl
  - Cyclohexyl amyl-phenyl
  - Cyclohexyl-benzyl
  - Xylyl-cyclohexyl
  - Lauryl-phenyl cyclohexyl
  - Cetyl-phenyl cyclohexyl
  - Phenyl-methylcyclohexyl
  - Phenyl-amylcyclohexyl
  - Xylyl-methylcyclohexyl
  - Benzyl-cyclohexyl

- Benzyl-methylcyclohexyl
- Methylcyclohexyl-amyl
- Methylcyclohexyl-lauryl

II. Oxygen-containing organic radicals

- A) Of the ether type, e. g.
  - Methoxy-phenyl
  - Ethoxy-phenyl
  - Lauroxy-phenyl
  - Cyclohexoxy-phenyl
  - Phenoxy-phenyl
  - Phenoxy-naphthyl
  - Phenoxy-cyclohexyl
  - Phenoxy-ethyl
  - Phenoxy-lauryl
  - Phenoxy-cetyl
  - Amyl-phenoxy-ethyl
  - Phenyl-ethoxy-ethyl
  - $-C_2H_4-O-C_2H_4-$ (derived from di-ethylene glycol)
- B) Of the ketone type, e. g.
  - Acetyl-phenyl
  - Acetyl-xenyl
  - Acetyl-naphthyl
  - Acetyl-lauryl
  - Acetyl-cetyl
  - Acetyl-cyclohexyl
  - Acetyl-naphthenyl
  - Lauroyl-phenyl
  - Benzoyl-phenyl
  - Benzoyl-naphthyl
  - Benzoyl-cyclohexyl
  - Benzoyl-naphthenyl
  - Benzoyl-lauryl
  - Benzoyl-cetyl
  - Stearoyl-phenyl
  - Stearoyl-xenyl
  - Stearoyl-naphthyl

Stearoyl-amyl
Stearoyl-lauryl
Stearoyl-cetyl
Stearoyl-phenoxyphenyl
Stearoyl-cyclohexyl
Stearoyl-naphthenyl
Naphthenoyl-phenyl
Naphthenoyl-xenyl
Naphthenoyl-naphthyl
Naphthenoyl-amyl
Naphthenoyl-lauryl
Naphthenoyl-cetyl
Naphthenoyl-phenoxy-phenyl
Naphthenoyl-cyclohexyl
Phenoxybenzoyl-phenyl

C) Of the ester type, i. e.

Derived from hydroxy-esters by the removal of the hydroxyl group, as for example, by the removal of the hydroxyl group from the following esters:

1) Esters of hydroxy acids, e. g.

Esters formed by substitution of any of the "R" groups listed above for one or more of the acid hydrogen atoms of the following acids:

Lactic acid
Hydracrylic acid
Citric acid
Tartaric acid
Ricinoleic acid
Hydroxy-stearic acid
Hydroxy-chlorstearic acid
Cyclohexanol carboxylic acids
Hydroxybenzoic acids
Salicylic acid
Hydroxy-phthalic acids
Hydroxy phenyl-benzoic acids
Naphthol carboxylic acids

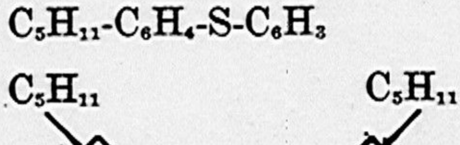
2) Esters of polyhydroxy alcohols and phenols in which at least one hydroxyl group remains unsubstituted, for example—esters formed by esterifying the following so that at least one hydroxyl group remains in the resulting ester:
   - Ethylene glycol
   - Diethylene glycol
   - Propylene glycols
   - Butylene glycols
   - Poly-hydroxy benzenes
     - Dihydroxy-benzenes
       - Resorcinol
       - Catechol
       - Hydroquinone
     - Tri-hydroxybenzenes
       - Pyrogallol

III. Sulphur-containing radicles, e. g.

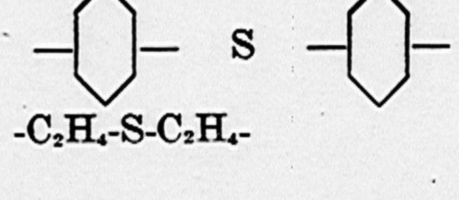
any of the examples of oxygen-containing radicles in which at least one oxygen atom has been replaced with sulphur. Specific examples of sulphur-containing radicles are:

A) Of the thio-ethyl type

Thio-phenoxy-phenyl (C_6H_5-S-C_6H_4-)
Thio-phenoxy-ethyl (C_6H_5-S-C_2H_4-)
Thio-lauroxy-phenyl ($C_{12}H_{25}$-S-C_6H_4-)
Thio-t-amyl-phenoxy-phenyl
(C_5H_{11}-C_6H_4-S-C_6H_4-)
Thio-t-amyl-phenoxy t-amyl-phenyl



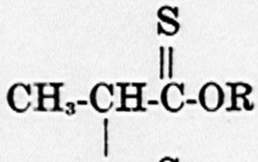
$C_5H_{11}\text{-}C_6H_4\text{-}S\text{-}\overset{\diagup C_5H_{11}}{C_6H_3}$



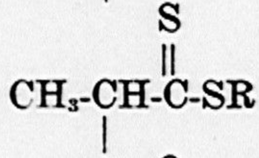
-C_2H_4-S-C_2H_4-

B) Of the thio-ketone type
- Thio-acetyl-phenyl
- Thio-benzoyl-phenyl
- Thio-stearoyl-phenyl
- Thio-naphthenoyl-phenyl
- Thio-stearoyl-cyclohexyl

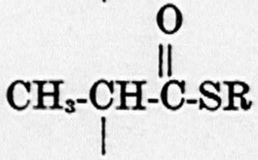
C) Of the thio-ester type, e. g.
Derived from hydroxy-esters of thio-carboxylic acids. Examples of such radicles are:



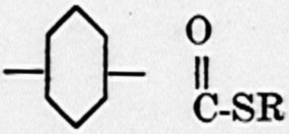
$$\mathrm{CH_3{-}\underset{|}{C}H{-}\overset{\overset{S}{\|}}{C}{-}OR}$$

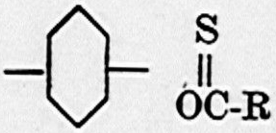


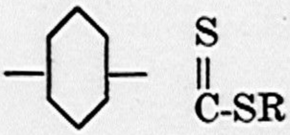
$$\mathrm{CH_3{-}\underset{|}{C}H{-}\overset{\overset{S}{\|}}{C}{-}SR}$$

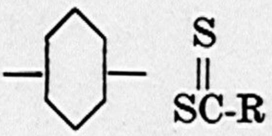


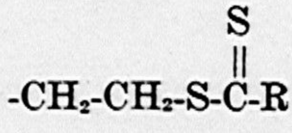
$$\mathrm{CH_3{-}\underset{|}{C}H{-}\overset{\overset{O}{\|}}{C}{-}SR}$$







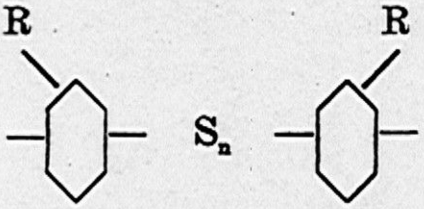




$$\mathrm{{-}CH_2{-}CH_2{-}S{-}\overset{\overset{S}{\|}}{C}{-}R}$$

Where R is methyl, ethyl, butyl, amyl, lauryl, stearoyl, naphthenyl, etc.

D) Radicles derived from hydroxy-organic polysulphides by removal of the hydroxy group, e.g.



where R is hydrogen or an alkyl or cyclo-alkyl group

$R\text{-}S_n\text{-}R'\text{-}$

where R is an alkyl or cyclo-alkyl group and R′ is an alkylene group.

The metals which, for example, are included within the scope of M in the foregoing formula are such as the following:—

Table No. 2

Calcium
Magnesium
Barium
Beryllium
Strontium
Aluminum
Chromium
Zinc
Cadmium
Cobalt
Nickel
Tin
Silver
Copper

The metals of Group II of the Periodic Table will be found useful and of these the alkaline earth metals are preferable.

The acid forming elements Y in the foregoing formula include the following for example:—

Table No. 3

Phosphorus
Arsenic
Sulphur
Selenium
Silicon
Boron

A list of examples of metallic alcoholates and thio-alcoholates which are included among the detergent materials contemplated hereby is as follows:—

Table No. 4

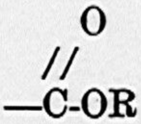
Aluminum butyl alcoholate
Calcium cetyl alcoholate
Calcium ceryl alcoholate
Calcium myricyl alcoholate
Calcium alcoholate of naphthenyl alcohol
Calcium alcoholate of cholesterol
Calcium alcoholate of naphthenoyl-benzyl alcohol
Calcium alcoholate of naphthenyl-cyclohexanol
Calcium alcoholate of ceryl-cyclohexanol
Calcium alcoholate of naphthenyl-thio-benzyl alcohol (calcium naphthenyl-benzyl mercaptide)
Calcium mercaptide of thio-cholesterol

In order to be sufficiently oil-soluble, the metallic phenates require a solubilizing group. In other words, they must be derived from substituted phenolic compounds which contain substituent groups which have the effect of making the resultant metallic phenates sufficiently oil-soluble for the purpose.

Representative examples of solubilizing groups which may be utilized as oil-solubilizing substituents in phenates are the following:—

Table No. 5

Lauryl
Cetyl
Carnaubyl
Ceryl
Montanyl
Myricyl
Naphthenyl
Naphthenoyl
Stearoyl



$$—\overset{\overset{O}{/\!/}}{C}\text{-OR}$$

where R is lauryl, cetyl and ceryl.

In addition to the foregoing specific examples which are preferred, it should be noted that any of the groups given in previous Table No. 1 (other than those of class B, and preferably those containing cyclo-alkyl alkylated cyclo-alkyl, and long-chain alkyl groups) may be used as solubilizing groups on the phenates.

Reference may be had to the following U. S. patents which disclose metallic phenates which may be used as the detergent component of the compounded lubricating composition of my invention:—2,197,833 and 2,228,661.

Additional specific examples of such stable oil-soluble metallic phenates are as follows:—

Table No. 6

Aluminum cetyl phenate
Calcium cetyl phenate
Calcium lauryl phenate
Magnesium phenate of lauryl salicylate
Calcium lauryl chlorphenate
Magnesium phenate of lauryl chlorsalicylate
Calcium phenate of di(tertiary amyl hydroxyphenyl) sulphide

The carboxylic acid salts useful as detergents may be those derived from:—

Table No. 7

(a) Fatty acids such as the following:
    stearic
    palmitic
    lauric
    dichlorstearic
    oleic

(b) Cycloaliphatic acids, such as:
    naphthenic acids
    alkylated hexahydrobenzoic acids
        lauryl hexahydrobenzoic acid
        cetyl hexahydrobenozic acid
    alkylated phthalic acids
        lauryl phthalic acids
        dilauryl phthalic acids
        cetyl phthalic acids
        dicetyl phthalic acids

(c) Aromatic substituted fatty acids, such as:
    phenyl stearic acid
    tolyl stearic acid
    xylyl stearic acid
    xenyl stearic acid
    naphthyl stearic acid
    phenoxy phenyl stearic acid
    benzoyl phenyl stearic acid
    hydroxy phenyl stearic acid
    chlor phenyl stearic acid
    phenyl palmitic acid

(d) Aliphatic and cycloaliphatic substituted aromatic acids such as the following:
    dilauryl benzoic acid
    naphthenyl benzoic acid
    dicyclohexyl benzoic acid
    di(methyl cyclohexyl) benzoic acid

naphthenoyl benzoic acid
stearoyl benzoic acid
monoceryl ester of phthalic acid

Specific examples of salts of carboxylic acids are the following:

Table No. 8

Calcium dichlor stearate
Calcium naphthenate
Cobalt naphthanate
Nickel naphthanate
Stannous naphthenate
Calcium phenyl stearate
Calcium chlor phenyl stearate
Cobalt chlorphenyl stearate
Magnesium dilauryl benzoate
Magnesium stearoyl benzoate
Magnesium naphthenoyl benzoate
Magnesium salt of mono-ceryl ester of phthalic acid

Examples of salt-esters of phosphorus acids are any of those in which the metals are selected from Table No. 2, and the organic radicles are selected from Table No. 1, for example, the ester radicle may be derived from the following alcohols and phenols:

Table No. 9

Lauryl alcohol
Cetyl alcohol
Ceryl alcohol
Methyl cyclohexanol
Amyl cyclohexanol
Di-amyl cyclohexanol
Lauryl cyclohexanol
Decahydro- -naphthol
Sterols, e.g. cholesterol

Borneol
Cetyl-benzyl alcohol
Wax-substituted benzyl alcohol
Stearoyl-benzyl alcohol
Naphthenoyl-benzyl alcohol
Lauryl-phenol
Cetyl-phenol
Ceryl-phenol
Wax-substituted phenol
Stearoyl phenol
Naphthenoyl phenol
Wax-substituted naphthols

Specific examples of salt-esters of phosphorus acids may be found in U. S. patents Nos. 2,228,658, 2,228,659, 2,252,984, and 2,252,985, and additional specific examples are as follows:

Table No. 10

The calcium salt of the di-lauryl ester of mono-thio phosphoric acid
The calcium salt of the di-cetyl ester of mono-thio phosphoric acid
The barium salt of the di-cetyl ester of mono-thio phosphoric acid
The zinc salt of the di-cetyl ester of mono-thio phosphoric acid
The calcium salt of the di(methyl-cyclohexyl) ester of mono-thio phosphoric acid
The zinc salt of the di(methyl-cyclohexyl) ester of mono-thio phosphoric acid
The calcium salt of the di(amyl-cyclohexyl) ester of mono-thio phosphoric acid
The zinc salt of the di(amyl-cyclohexyl) ester of mono-thio phosphoric acid
The calcium salt of the di-cholesteryl ester of mono-thio phosphoric acid

The zinc salt of the di-cholesteryl ester of mono-thio phosphoric acid
The zinc salt of the di-bornyl ester of mono-thio phosphoric acid
The calcium salt of the di-naphthenyl ester of mono-thio phosphoric acid
The calcium, barium, strontium and magnesium salts of the mono- and di-: amyl-cyclohexyl, di-amyl-cyclohexyl, naphthenyl, bornyl, cholesteryl, naphthenoyl-phenyl, stearoyl-phenyl, ceryl, and miricyl esters of phosphoric acid.

Specific examples of salts and of salt-esters of phosphonic and thio-phosphonic acids and of salts of phosphinic and thio-phosphinic acids may be found in U. S. patents Nos. 2,228,658, 2,228,659, 2,252,984, and 2,252,985. Additional specific examples are:—

Table No. 11

The calcium and magnesium salts of the mono-(amyl-cyclohexyl) esters of lauryl-phenyl, stearoyl-phenyl, naphthenoyl-phenyl, and ceryl phosphinic and thio-phosphinic acids.

Specific examples of the salt-esters of sulphuric acid and thio-sulphuric acid are as follows:

Table No. 12

The calcium and magnesium salts of the mono- : cetyl, ceryl, lauryl-cyclohexyl, naphthenyl, naphthenoyl-benzyl, and stearoyl-benzyl esters of sulphuric and thio-sulphuric acids.

Specific examples of salts of sulphonic and sulphinic acids are as follows:

Table No. 13

The calcium, barium, strontium, aluminum, chromium, and magnesium salts of: "mahogany acids", (e.g. the product obtained by fuming sulphuric acid treatment of "white oils") and the calcium barium, strontium and magnesium salts of the corresponding sulphinic acids (e.g. the acids produced by neutral or alkaline reduction of the acid chlorides of mahogany acids).

Specific examples of salt-esters of arsenic-containing acids are as follows:

Table No. 14

The calcium, barium, strontium, magnesium, zinc, and cadmium salts of the di-: amyl-cyclohexyl, di-amyl-cyclohexyl lauryl-cyclohexyl, cetyl-cyclohexyl, naphthenyl, bornyl, cholesteryl, ceryl and miricyl esters of arsenic acid (H_3AsO_4), arsenious acid (H_3AsO_3) and thio-arsenic acids.

Specific examples of salt-esters of selenium-containing acids are as follows:

Table No. 15

The calcium, barium, strontium, magnesium, zinc and cadmium salts of:

(a) The mono-: cetyl, ceryl, miricyl, lauryl-cyclohexyl, di-lauryl-cyclohexyl, cetyl-cyclohexyl, naphthenyl, bornyl, and cholesteryl esters of selenic acid (H_2SeO_4), selenious acid (H_2SeO_3); and

(b) The mono- and di-: amyl-cyclohexyl, di-amyl-cyclohexyl, lauryl-cyclohexyl, cetyl-cyclohexyl, bornyl, cholesteryl, lauryl, cetyl, ceryl and miricyl esters of mono-seleno-phosphoric acid and di-seleno-phosphoric acids.—

Remarks

The additional art cited by the Examiner has been carefully considered certain of the claims amended, and others cancelled in effort to place the application in condition for allowance; and in view of these changes and the remarks as herein presented, reconsideration of the application is respectfully solicited.

In the first paragraph of the above-identified Office action, the Examiner rejects certain of the claims in the use of the term "characterizing amount" as being indefinite. The quantities of the "characterizing addition agents" range from about 0.1% to 4% based upon the weight of the total composition, as pointed out in lines 22-26 of page 13 of the specification. On page 14, lines 1-4, a preference is indicated for the narrower range of 0.5 to 2.5%. In view of this disclosure, it is believed that the term "characterizing amount" is neither vague nor indefinite. If, however, the Examiner still believes that such terminology is vague and indefinite in view of the specification, will he please indicate the manner in which these claims may be amended to overcome the rejection in this regard.

Claims 16, 18, 20 and 21 have been rejected as being too broad and indefinite in the use of the term "corrosion inhibitor". It is believed that to restrict the term or to eliminate it entirely from the claims would render the coverage of the invention, to which the applicant is entitled, insufficient. The use of corrosion inhibitors in conjunction with the characterizing addition agent is as much a part of the entire scope of the invention as is the use of other addition agents, such as halogenated organic compounds, detergents, pour-point depressors, and the like in combination therewith. Corrosion inhibitors as such are well known and widely used in lubricants. However, the applicant is the first to teach the use of corrosion inhibitors in combination with another inhibitor of the type with which this invention is primarily concerned. None of the art cited by the Examiner shows the particular combination employed by the applicant. It is therefore believed that the use of the term corrosion inhibitor in the claims is not indefinite.

Certain of the claims have been rejected on the ground of insufficient disclosure as to the detergents claimed. This disclosure which according to the unamended specification was to be found in a copending application of Carl F. Prutton has been amended to overcome this informality as pointed out by the Examiner.

The Examiner in his rejection of certain of the claims relies upon the patent to Cook which discloses a 2-step process for the preparation of zinc salts of the reaction product of capryl alcohol with phosphorus pentasulphide. The applicant's process as now defined by the claims covers the product resulting from the simultaneous admixture of capryl alcohol, phosphorus pentasulphide, and zinc oxide. The claims 1-8 inclusive which have been rejected on Cook have been either cancelled or amended so as to more clearly define the applicant's product in contradistinction to that of Cook.

The rejection of the same claims 1-8 on the patent to Salzberg is for the reasons above expressed with reference to the Cook patent likewise overcome. Furthermore, Salzberg is concerned with aliphatic groups containing 10 or more carbon atoms, whereas the applicant is concerned with capryl alcohol which is a secondary 8 carbon alcohol.

Claims 9-24 have been rejected over the Cook and Salzberg patents in view of the patents to Wilson and McNab. As pointed out by the Examiner, the Wilson and McNab patents disclose the use of corrosion inhibitors in combination with detergents. However, it is respectfully submitted that although the basic suggestion of the combined use of corrosion inhibitors and detergents may be old, the applicant has advanced the art materially by pointing out that certain detergents i.e. those selected from "the class consisting of oil soluble organic compounds containing a metal atom directly attached to an element of the class consisting of oxygen and sulphur" cooperate to produce a result which is above and beyond that which may be expected as a result of reading the Cook and Salzberg patents in combination with the Wilson and McNab patents. Results obtained from the testing of various combinations of these detergents so described by the applicant and the characterizing addition agent of this invention are reproduced in tables beginning on page 8a and concluding on page 8c. For example on page 8b Table B, it will be observed that after 60 hours of operation untreated S.A.E. 30 solvent refined Mid-Continent motor oil having a V.I. of 85 showed a bearing loss of 117 mgms., .75% of zinc oxide, capryl dithiophosphate in the same oil at the end of the same time, showed a bearing loss of 26 mgms., 0.4% of calcium salt of petroleum sulphonic acid in the same oil showed a bearing loss of 341 mgms. at the end of the same period of time. Sample No. 6 which was a combination of zinc dicapryl dithiophosphate and the calcium salt of petroleum sulphonic acid at the end of 90 hours, i.e. 30 hours more than the previous ratings, showed a bearing loss of only 21 mgms. which is less than any of the previous examples cited. This shows conclusively, that these materials do cooperate to produce a result which is far superior to what would be expected from the teachings of the prior art.

Under the circumstances therefore it is respectfully submitted that the application is now in condition for allowance and early action to that effect will be greatly appreciated.

Respectfully submitted,
Oberlin, Limbach & Day
Attorneys

* * * *

Responsive to amendment filed Jan. 12, 1945.

Claims, 1, 9, 10, 11, 16, 17, 18, 21, 22, 23 and 24 are rejected for reasons stated in the first paragraph of last office action. The term "characterizing amount" is vague, broad and indefinite for applicant fails to indicate what properties are imparted to a lubricating oil by the addition of a reaction product of zinc salt of dicapryl dithiophosphate. Applicant should either indicate the amount of oil improving agent used or state that amounts sufficient to impart certain desired results are used.

The rejection of claims 16, 18, 20 and 21 for reasons stated in the second paragraph of last office action is deemed sound and is adhered to.

Claims 1, 5 and 9-24 are rejected over the Cook patent of record which discloses the use of zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide as a lubricating oil improving agent, as well as the comibnation of this zinc salt reaction product with other lubricating oil additives such as alkaline earth metal salts of petroleum sulfonates (well known detergent agents) and well known oil-soluble organic corrosion inhibitors.

The process limitation, that is, claiming the reaction product by simultaneously reacting the zinc oxide, capryl alcohol and phosphorus pentasulfide does not amount to patentable subject matter since the final product is exactly the same whether it is produced in a stepwise manner or not. The alleged invention does not reside in the process of producing the product but in the product itself. Furthermore, all examples as to the method of producing this product are directed to stepwise limitation, the same as Cook uses. The only reference directed to a one-step process is incidentally disclosed on page 6 of the specification and this does not show that an improved or different product is obtained over a stepwise method in producing zinc dicapryl dithiophosphates.

The rejection of claims 9-24 for reasons stated in the last paragraph of last office action is also adhered to.

A definite issue having been reached this action is herewith made FINAL.

/s/ P. M. Nash
Examiner

CGP/n

* * * *

Hon. Commissioner of Patents
Sir:

In order to place the application in better condition for an appeal, applicant hereby amends as follows:—

Claims 1, 9, 10, 11, 16, 17, 18, 21, 22, 23 and 24, lines 1 and 2 of each, cancel the words "a characterizing amount" and insert in lieu thereof —an amount sufficient to impart corrosion inhibiting properties—.

Remarks

The foregoing amendment to certain of the claims has been made for the purpose of placing the claims in better condition for appeal and at the suggestion of the primary Examiner in the first principal paragraph of his Office action dated March 30, 1945, entry of the foregoing amendment is respectfully requested.

Respectfully submitted,
Oberlin, Limbach & Day
Attorneys

* * * *

To the Commissioner of Patents,
Sir:

I hereby appeal to the Board of Appeals from the decision of the principal Examiner in the matter of my above-identified application for Letters Patent, which on the 30th day of March, 1945, was finally rejected. The following are the points of the decision on which the appeal is taken.

1. The Examiner erred in rejecting the claims.
2. The Examiner erred in not allowing the claims.
3. That the Examiner erred in rejecting claims 16, 18, 20 and 21 as too broad and indefinite as to the use of the term "corrosion inhibitor".
4. That the Examiner erred in rejecting the claims over the Cook patent 2,344,392.

5. That the Examiner erred in rejecting the claims over the Cook and Salzberg patents in view of the patents to Wilson and McNab.

Oral hearing is respectfully requested.

Respectfully submitted,
Oberlin, Limbach & Day
Attorneys

Cleveland, Ohio
September 24, 1945

ABSTRACTED FILE WRAPPER & CONTENTS IN RE U. S. ABANDONED SERIAL NO. 401,960, ELMER WILLIAM COOK, ET AL.

* * * *

Specifications

TO ALL WHOM IT MAY CONCERN:

Be it known that we, Elmer William Cook and William D. Thomas, Jr., citizens of the United States and residents of New York, New York County, New York and Stamford, Fairfield County, Connecticut, respectively, have invented certain new and useful improvements in

CRANKCASE LUBRICANTING OIL COMPOSITIONS

of which the following is a specification:

This invention relates to lubricating oils of the type known as crankcase oils; i.e., hydrocarbon oils of relatively low viscosity and pour point such as are used in the crankcases of internal combustion engines. More particularly, the invention relates to the newer classes of crankcase oils for truck, bus, airplane and marine gasoline and diesel engines of the type known as "heavy duty" oils, which contain added chemicals that increase their effective service life under heavy operating conditions. A principal object of the invention resides in the provision of a crankcase lubricant of this type containing a class of compounds having both detergent and anti-oxidant properties, whereby the formation of hard sludge deposits is inhibited. A further object resides in the provision of an inhibitor of this class which is of the metal salt type, but which has little or none of the undesirable oxidizing action on hydrocarbon oils that is a characteristic of many metal salt inhibitors.

We have discovered that the above and other objects are accomplished when oil-soluble heavy metal salts of dithiophosphoric acid esters are added to crankcase oils in relatively small amounts; i.e. in concentrations of about 0.1% to 3% of the heavy metal salt, based on the weight of the oil. We have found that these compounds are excellent detergents for the prevention of sludge deposits in the engine, and that they also possess extremely important non-oxidizing or anti-oxidant properties when compared with other metal salt detergents that have previously been used. Thus, for example, the oxygen uptake of a sample of paraffin base crankcase oil containing 0.3% of barium diamyl dithiophosphate was 300 cc. when blown with a stream of oxygen at 170° C. for 24 hours in a glass system. The corresponding figure for the same oil containing no detergent or other addition agent was 280 cc. while another sample containing 0.2% of barium naphthenate, a conventional detergent, was 525 cc.

Our invention in its broader aspects therefore consists in the addition to lubricating oils of the type of hydrocarbon crankcase oils of a heavy metal salt of a dithiophosphoric acid ester, either alone or in admixture with other ingredients, in amounts sufficient to exert sludge dispersing properties under heavy duty service conditions but without substantial increase in oxygen absorption and consequent sludge formation in the oil. The formation of sludge in crankcase oils is intimately related with the production of corrosive oxidation products of the type of organic acids, and analytical determination of these decomposition products is usually accomplished by measuring the naphtha insoluble fraction of the oxidized oil. This feature of our invention can therefore be expressed by stating that the heavy metal salts in question do not increase the naptha-insoluble value of the oil, and in fact these compounds usually bring about a marked reduction in this value. The importance of this property in our new detergents will now be explained.

The constant increase in the power output of internal combustion engines during the past few years has imposed increasingly severe requirements on the crankcase oils therein. The operating temperatures are higher and therefore the lubricating oils decompose more rapidly, forming complex and objectionable oxidation and decomposition products. Under the high temperature conditions obtaining in the engine these decomposition products polymerize to form lacquer-like deposits on and between the moving parts, causing these parts to stick. Even larger quantities of polymerization products remain dispersed in the partly oxidized crankcase oil, and are rapidly precipitated in the form of sludges when the engine cools or when fresh oil is added to the engine. These precipitated sludges become caked on the heated metal surfaces and cut down the effective life of the engine.

A number of oil-soluble detergents of the type of metallic soaps, phenolates and alcoholates have been proposed and used in crankcase oils to dissolve or disperse the sludge and prevent lacquer deposits and stuck piston rings. Unfortunately, however, the great majority of these detergent substances increase the rate of oil oxidation in a manner somewhat analogous to the accelerating action of the various metallic soaps or "driers" on the oxidation of varnish films, and their presence results in an increased concentration of acidic oxidation products in the oil. Increased quantities of these acidic oxidation products in the oil creates in turn an even more serious difficulty, for they attack and corrode the alloy connecting rod bearings that have replaced the conventional babbitt bearings to a great extent in internal combustion engines. Copper-lead alloy bearings are perhaps the most widely used bearings at present, but silver-cadmium, nickel-cadmium and other bearings of this type are also used, and all these bearings are rapidly attacked by the acidic oxidation products.

The importance of our present invention is apparent from the foregoing discussion. We have found a class of addition agents for crankcase oils which are not only excellent detergents to prevent the accumulation and deposition of sludges on the metal surfaces of the engine, but which have no accelerating action on the oxidation and subsequent polymerization of the oil and actually reduce the amount of free acids therein. In other words, we have discovered that small quantities of oil-soluble heavy metal salts of dithiophosphoric acid esters, when present in a crankcase oil, will effectively prevent the deposition of insoluble sludges while also inhibiting to a marked extent the oxidation of the oil.

It is interesting to compare the results obtained by the incorporation of small amounts of the heavy metal salts of dithiophosphoric acid esters in crankcase oils with the action of mixtures of dissimilar chemicals that have been added to oils in an attempt to accomplish the same purpose. Detergents such as calcium stearate, calcium naphthenate and the like have previously been used in admixture with anti-corrosion agents such as triphenyl phosphite and sulfurized sperm oil that have no detergent properties. Although each of these two classes of materials will perform its function in a lubricating oil the two separate chemicals do not cooperate to produce a satisfactory anti-corrosion and detergent action when used together. The function of a corrosion inhibitor is to cover the bearings and other corrodable metal surfaces of the automobile engine with a passivating film that prevents corrosion of the metal by the organic acids and other corrosive oxidation products in the oil. On the other hand a sludge inhibitor or detergent, as its name implies, operates to remove adhering solids from the metal parts of the engine and thus to produce a clean metal surface. Consequently, the use of a corrosion inhibitor in admixture with the detergents heretofore employed has proven to be ineffective over any extensive period of time, as each chemical tends to offset the action of the other.

As has been stated, our invention in its broader aspects includes the addition to crankcase oils of small amounts of any heavy metal salt of an ester of dithiophosphoric acid that is soluble therein. We have discovered that the dithiophosphoric acid diesters of alcohols of the aliphatic, aromatic and cycloaliphatic series will form heavy metal salts that are soluble in crankcase oils to the extent of at least 0.1-3% when each alcohol radical contains at least 5 carbon atoms. For reasons that subsequently will be explained, our preferred detergents are the heavy metal salts of dialkyl dithiophosphates in which each alkyl group contains 8, 10, 12, 14 or more carbon atoms, although good results have been obtained with heavy metal salts of dicyclohexyl and diaryl dithiophosphates. Moreover, we have succeeded in incorporating substantial amounts, even greater than 3%, of barium and other heavy metal salts of the lower dialkyl dithiophosphates such as those of propyl, butyl and amyl alcohol into lubricating oils by first admixing or dissolving them in metal salts of 2,4-diamylphenol sulfides having the formula

$$(C_5H_{11})_2C_6H_2(OM).S.(MO)C_6H_2.(C_5H_{11})_2$$

in which each M may be a monovalent metal or the two M's may represent one atom of a divalent metal. Obviously salts of other 2,4-dialkylphenol sulfides such as those containing from 5-21 carbon atoms in one or more of the alkyl groups may be employed for the same purpose as is pointed out in our patent No. 2,249,626 dated July 15, 1941.

Although dithiophosphate ester salts of any heavy metal may be used in practicing our invention we prefer to employ the salts of zinc, cadmium, aluminum, tin, and lead and particularly the salts of the alkaline earth metals which are magnesium, calcium, barium and strontium. The salts of the above group of metals with dithiophosphoric acid esters are sufficiently oil-soluble to present no serious difficulties in practicing the invention, and they all possess excellent detergent properties. While similar detergent properties are possessed by salts of heavy metals other than those listed above, we are aware of no advantage to be obtained by their use. On the other hand, the alkaline earth metal salts are particularly advantageous both because they are quite soluble in hydrocarbon oils and insoluble in water and because they possess outstanding detergent properties.

The dialkyl esters of dithiophosphoric acid, when used in the form of their heavy metal salts, constitute the preferred class of compounds for use in practicing our invention, as these dialkyl esters possess excellent detergent properties. This is particularly true of the dioctyl and dilauryl esters of dithiophosphoric acid which, when used in the form of their alkaline earth metal salts, are the outstanding members of the class in question. The results obtainable with small amounts of these compounds are shown in the following examples as illustrative of the invention as a whole, and it will be noted that in all cases the naphtha-insoluble value of oils containing them is much lower after the test period than the corresponding values for the untreated oils.

Although crankcase oils under service conditions will contain only small amounts of the heavy metal salts of the present invention, it should be understood that for commercial reasons the appended claims are not limited to hydrocarbon oils containing such small quantities except where such limitations actually appear in the claims. It is an important advantage of the invention that the preferred heavy metal salts included therein are extremely soluble in hydrocarbon oils, and that stock solutions of high concentration can be prepared for storage and shipping. The barium, calcium and other alkaline earth metal salts of dioctyl and dilauryl dithiophosphates, for example, are soluble in ordinary 10-W grade lubricating oil to the extent of 50-80% at 25° C., and strong solutions of this order of concentration may readily be prepared and shipped to oil refineries and other distribution centers for blending with lubricating oils in the small proportions needed for use.

The invention will be further illustrated in detail by the following specific examples, which present results obtained with representative heavy metal salts of dialkyl and diaryl dithiophosphates. It should be understood, however, that the invention in its broader aspects is not limited to these examples, and that the results set forth therein are therefore to be regarded as indicative of similar advantages obtainable by the invention as a whole.

Example 1

Two samples of solvent refined Pennsylvania (paraffin base) lubricating oil of 10-W grade were subjected to the Underwood oxidation test with and without the addition of detergents. This test consists in heating 1500 cc. of the oil under test to 325° F. and continuously spraying a portion of the heated oil against a 2″x10″ freshly sanded copper strip and two freshly sanded bearings to be tested for corrosion for five hours while permitting free circulation of air through the apparatus. Samples of the oxidized oil were then examined for specific gravity, neutralization number, and naphtha insoluble and the bearings under test were weighed to determine loss by corrosion. The results were as follows:

Table I

Sample #1	A.P.I. Gravity	Naphtha Insolubles %	Bearing Loss - Mg Ag-Cd	Cu-Pb	Neut. No.
Initial A.P.I. Gravity = 31.1)	24.5	0.28	973	82.4	9.41
+ 0.2% Barium dilauryl dithiophosphate	29.6	0	1	0.3	1.26
+ 0.2% Calcium stearate	23.1	0.62	996	124	11.90
Sample #2					
Initial A.P.I. Gravity = 30.4)	27.1		411	46	
+ 0.1% Iron naphthenate	21.9		940	32	
(0.1% Iron naphthenate + (0.4% Barium dilauryl dithiophosphate	29.9	0	13	2	

Iron naphthenate is known to be a powerful promoter of oxidation and decomposition in lubricating oils. Compounds similar to this substance are usually present in engines containing oxidized oil as a result of the attack on the metal by the acidic oxidation products. The effectiveness of the heavy metal salts of dithiophosphoric acid esters in overcoming the decomposition caused by iron salts of naphthenic acids is another important advantage of the present invention.

Example 2

Another widely used test for accelerated oxidation in crankcase oils is known as the Catalytic Indiana test. The apparatus consists of a constant temperature bath maintained at 341° F. in which a number of large glass test tubes are immersed. 300 cc. samples of the oil under test are poured into these tubes and air at the rate of 10 liters per hour is bubbled through the oil. In order to reproduce the conditions existing in the crankcase of an engine weighed strips of copper-lead alloy are suspended in the oil samples. As metallic surfaces, particularly copper, greatly accelerate the rate of oxidation and decomposition of the oil in the presence of oxygen this is an important factor in the test. Bearing corrosion rates can also be determined by again weighing the strips after 70 hours immersion, which is the usual test period.

Heavy metal salts of representative dithiophosphoric acid esters were tested against crankcase oils of standard domestic types by the above procedure, with the following results:

Table II

Oil Base Stock, Solvent Extracted Pennsylvania Oil, 10-W Grade

	A.P.I. Gravity Oxidized Oil	Naphtha Insol- ubles %	Bearing Loss-Mg Cu-Pb	Neut No.
Oil	23.5	17.47	215	4.63
Oil + 0.3% Barium dicapryl dithiophosphate	27.3	3.02	+16	3.19
Oil + 0.3% Barium di-n-octyl dithiophosphate	27.6	1.83	+10	3.38
Oil + 0.3% Barium dilauryl dithiophosphate	27.4	1.43	+7	3.50
Oil + 0.3% Barium dioctadecyl dithiophosphate	26.9	2.72	1	4.13
Oil + 0.3% Barium di(p-tert-amylphenyl)dithiophosphate	27.7	2.32	+2	2.95
Oil + 0.3% Barium di(2,4-diamylphenyl)dithiophosphate	27.3	2.02	+10	3.24
Oil + 0.3% Zinc dibutyl dithiophosphate			+7	
Oil + 0.4% Zinc dilauryl dithiophosphate			4	

* * * *

Hon. Commissioner of Patents,
Sir:

Please amend the claims as follows:

Cancel claims 1 and 5 without prejudice to applicant's right to include the subject matter thereof in a divisional or continuation application.

Claims 16, 18, 20 and 21, last line of each, change the period following the word "inhibitor" to a comma, and add —selected from the class consisting of phenolic compounds.—

Remarks

The foregoing amendment is made for the purpose of removing the Examiner's objection to the use of the term "corrosion inhibitor" as being too broad and indefinite. The claims containing such phrase have been modified to define the "corrosion inhibitor" as a phenolic compound.

The basis for such definition is found on page 12 of the specification where several examples of phenolic compounds are mentioned.

The amendment is also entered for the purpose of placing the claims in better condition for appeal.

Entry of the foregoing amendment is respectfully solicited.

Respectfully,
Oberlin, Limbach & Day
Attorneys

January 15, 1947

* * * *

Examiner's Report

Applicant has filed a request for consideration and report on the affidavit under Rule 75 filed with his brief on appeal on January 16, 1947. This affidavit was refused consideration by the Examiner for the reason that it was not seasonably presented. Applicant's request for consideration is accompanied by an affidavit purporting to show why the Rule 75 affidavit was not earlier presented. The Board of Appeals has remanded the case to the Primary Examiner for such disposition of said papers as he deems proper.

The affidavit under Rule 75 attempts to antedate the Cook patent of record. The showing as to why the affidavit was not earlier presented gives as the reason that in the Office Action of July 22, 1944, in which the Cook patent was first cited a patent to Salzberg was also cited, and claims 9 to 24 were rejected "over the Cook and Salzberg patents in view of the patents to Wilson and McNab." In the Office Action of March 30, 1945, the rejection on the Cook patent above was made for the first time. Affiant states that applicant's dates do not antedate the Salzberg patent and "so long as the Examiner, therefore, considered the Cook and Salzberg patents as references of equivalent weight, and combined them with the Wilson and McNab patents, there was no real reason for the filing of an affidavit under Rule 75." He further states that since the final rejection included a rejection on Cook alone for the first time, "There was no real reason why the applicant should have filed the affidavit under Rule 75 prior to the final rejection."

It is not believed that a satisfactory showing as to why the affidavit under Rule 75 was not filed earlier has been made. In the first place, even if it were assumed that there was no reason for filing the affidavit prior to the final rejection, it has not been shown why there was a delay of almost two years after the final rejection before the affidavit was filed. Furthermore, if applicant believed that the final rejection was premature, he could have petitioned to have finality of the rejection withdrawn. Still further, the fact that the rejection was first made on Cook and Salzberg in view of Wilson and McNab is not seen to be an adequate reason for failing to file the affidavit. If the Cook patent were removed as a reference, then the combination of Cook with Wilson and McNab would fall. The fact that the claims were also rejected on the Salzberg patent does not relieve applicant of the duty of seasonably antedating Cook. If the rejection on Salzberg should not be upheld, then the rejection involving Cook would still have to be dealt with. As stated in Ex parte Berg, 1906 C. D. 36.

"The practice of the Office . . . requires the applicant in the prosecution of his case to present at each step the best and all reasons why the claims should be allowed. If references can be overcome by affidavits, they should be presented at once for consideration by the Office, so that the case may, upon each action, take a decisive step toward completion."

It should be noted that the rejection on Cook and Salzberg in view of Wilson and McNab still stands (page 6 of Examiner's Statement). Accordingly, applicant's reasons for filing the affidavit now are no better than they were at the time the Cook patent was cited. It is not believed that applicant could successfully argue that the affidavit could be filed after decision by the Board, if the rejection should be reversed on Salzberg but affirmed on Cook.

Attention is also directed to Ex parte Remunder, 1910 C. D. 121. In this case the antedating affidavits were filed while the case was on appeal. Applicant's reason for delay in filing the affidavit was that, during the prosecution, numerous references had been cited by the Examiner without special reference to Smith (the reference in question) and that "the Examiner's Statement, in which the previously merely incidental or casual Smith reference leaps to the foremost place as a reference, is in the nature of a surprise."

The request for consideration of the affidavit was denied, the excuse for delay being held to be "manifestly inadequate."

This decision is considered even more applicable to the facts herein. In the instant case the Cook patent did not first leap to the foremost place in the Examiner's Statement and never was applied as a "merely incidental or casual" reference.

For the reasons indicated, the request for consideration of the affidavit under Rule 75 is denied.

/s/ P. M. Nash
Examiner.

* * * *

This is an appeal from the action of the Examiner finally rejecting claims 1, 5 and 9 to 24, inclusive.

Claim 1 is illustrative and reads as follows:

1. Lubricating composition containing a characterizing amount of the product obtained by the simultaneous admixture of capryl alcohol, phosphorus, pantasulphide, and zinc oxide.

The references relied upon are:

Salberg et al,	2,063,629,	Dec. 8, 1936,
Wilson,	2,280,419,	Apr. 21, 1942,
McNab,	2,289,795,	July 14, 1942,
Cook et al,	2,344,392,	Mar. 14, 1944.

Appellant does not contend that Cook et al do not disclose the composition of claims 1 and 5, and the Examiner has refused to consider the proposed affidavit under Rule 75 to antedate Cook et al.

The same applies to claims 9 to 24 as to the added detergents. There appears to be nothing of an inventive or novel character in the additional use of a halogen-bearing organic compound.

The Examiner has rejected claims 16, 18, 20 and 21 as too broad and indefinite with respect to "corrosion inhibitors." We regard the Examiner's position on these claims as correct, particularly if the corrosion inhibitor is contended to exert a critical and unobvious influence on the action of the zinc salt.

Salzberg et al are cumulative with Cook et al, when used in combination with McNab and Wilson. McNab uses an activator for the detergent, the activator being of the type employed by appellant. The same is true of Wilson.

The decision of the Examiner is affirmed.

In event of appeal attention is directed to *In re* Boyce, 32 C. C. P. A. 718, 144 Fed. (2d) 896, 1944 C. D. 609, 568 O. G. 568, 63 U. S. P. Q. 80, in regard to specifically including in the appeal notice *all* grounds of rejection in the Examiner's Statement not expressly overruled by the Board.

/s/ W. L. Redrow
Examiner-in-Chief
/s/ F. J. Porter
Examiner-in-Chief
/s/ B. H. Enkin
Examiner-in-Chief
(Acting)

BOARD
OF
APPEALS
March 31, 1947

Patented Oct. 28, 1941 2,261,047

UNITED STATES PATENT OFFICE

2,261,047

LUBRICANT

Peter A. Asseff, Cleveland, Ohio, assignor to The Lubri-Zol Corporation, Wickliffe, Ohio, a corporation of Ohio

No Drawing. Application July 28, 1941, Serial No. 404,361

14 Claims. (Cl. 252—35)

This invention relates as indicated to improved lubricants and more particularly to compounded lubricants which comprise a mineral lubricating oil base, the properties of which affecting its use as a lubricant will be improved by the inclusion therein of an addition agent.

Refined mineral lubricating oils while useful over a wide range of applications, are, nevertheless, not satisfactory for certain uses either on account of their tendency to deteriorate or on account of their inability to withstand high film pressures or otherwise perform the lubricating function required.

It is a principal object of this invention, therefore, to provide a compounded lubricant which while including a mineral oil base has properties which cannot be secured by the use of mineral oil alone.

Other objects of the invention will appear as the description proceeds.

To the accomplishment of the foregoing and related ends, said invention then comprises the features hereinafter fully described and particularly pointed out in the claims, the following description setting forth in detail certain illustrative embodiments of the invention, these being indicative, however, of but a few of the various ways in which the principal of the invention may be employed.

Broadly stated, the compounded lubricating composition of this invention may be defined as comprising a major amount of a hydrocarbon oil, such as mineral lubricating oil, and a minor amount of a zinc salt of an acidic reaction product obtained by reaction phosphorus pentasulphide with a cyclo-aliphatic alcohol.

In the foregoing broad statement of this invention the addition agent has been defined in terms of its mode of preparation and desirably and necessarily so on account of the difficulty in a more precise identification of the molecule characterizing the addition agent. As a matter of fact the addition agent is probably a mixture of different constituents and it is for this reason that the same is best defined, as above indicated, in terms of its mode of preparation. The addition agent may be prepared as indicated by reacting phosphorus pentasulphide with a cycloaliphatic alcohol in the preparation of an intermediate acidic reaction product and the zinc salt then prepared of such acidic reaction product.

Alternatively, the final product may be prepared in a single step or in concurrent steps by reacting together phosphorus pentasulphide the alcohol, and zinc oxide. In this case, it is probable that the reaction which forms the intermediate acidic reaction product, and the reaction of such acidic reaction product with zinc oxide to form the final salt, take place simultaneously.

The following is a list of cycloaliphatic alcohols which may be used in carrying out this reaction, and in such list, the specific examples are arranged substantially in the order of preference.

Methyl cyclohexanol
Ethyl cyclohexanol
Propyl cyclohexanol
Amyl cyclohexanol
Sterols, e. g. "Cosmol" (a cyclic alcohol derived from wool-fat)
Cyclohexanol
Methyl cycloheptanol
Methyl cyclopentanol
Cycloheptanol
Cyclopentanol

It is not necessary that the cycloaliphatic alcohol used for this purpose be a pure compound. It may consist of a mixture of cycloaliphatic alcohols and such mixtures are often particularly advantageous. An example of such material is the mixture of alcohols resulting from the hydrogenation of commercial cresol. The "methyl cyclohexanol" referred to hereafter is the product thus obtained. A mixture of alkylated cyclohexanols consisting principally of ethyl and propyl cyclohexanols has also been found particularly suitable for this purpose.

The cycloaliphatic alcohols which contain the cyclohexane nucleus, for example cyclohexanol and the alkylated cyclohexanols, are particularly useful cycloaliphatic alcohols for my purpose because they are commercially available and produce a product which has advantageous properties.

Of the cycloaliphatic alcohols in general those are preferred which have a total of less than ten carbon atoms. The alkylated cycloaliphatic alcohols are particularly suitable. In this case, it is best to have the alkyl group contain less than six and preferably less than four carbon atoms.

Since a specific example of the addition agents of the present invention which has been found of especial utility is the zinc salt of the acidic reaction product obtained by reacting phosphorus pentasulphide with methyl cyclohexanol, the details of the manufacture of such material will be given as illustrative of the mode of manufacture of the similar addition agents comprised within the broad statement of the invention, This process may be divided into two parts, i. e.

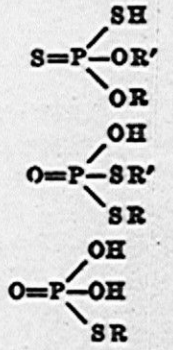
(*a*) The preparation of the acid ester, and
(*b*) The preparation of the metallic salt of such acid ester.

(*a*) Preparation of the Acid Ester

Five moles of methyl cyclohexanol and one mole of P_2S_5 were mixed with an amount of benzene equal in volume to the amount of cyclohexanol and placed in a flash equipped with a reflux condenser, a thermometer and a mechanical stir. The flask was placed in a water bath at approximately 100° C. and refluxed for four hours. The batch was then cooled to about 60° C. and washed once with an equal volume of water at 60° C. The benzene was removed from the wash material by distillation at atmospheric pressure. A typical batch in the preparation of the acid ester in the mode above defined was as follows:

Wt. of methyl cyclohexanol____grams___	500
Moles of methyl cyclohexanol__________	4.39
Weight of P_2S_5______________grams___	195.5
Moles of P_2S_5______________________	.88
C. c. of benzol used as solvent__________	500
C. c. of 60° C. H_2O used in washing_____	1000
Weight of product____________grams___	616
Temp. of reaction, °C:	
Bath temp________________°C__	100
Vapor temp_______________°C__	80
Time of reaction_____________hours___	4

It is believed that the reaction between phosphorus pentasulphide and the alcohol produces a mixture of compounds of the type represented by acid thiophosphate esters. For example:



where:

R is the cyclo-aliphatic radicle derived from the alcohol
R' is hydrogen or R

The final product, if this assumption is correct, would then consist of the salts derived from the acid ester by replacing the replaceable hydrogen of such esters with zinc.

(*b*) Preparation of the Metallic Salt

First method

The acid ester resulting from step (*a*) above was mixed with enough water to make a 15–20% mixture. This solution was made neutral to phenolphthalein with 30% aqueous caustic. The acid was neutralized at 80–90° C. To the soda-salt solution was added an aqueous solution of zinc chloride. The reaction mixture was stirred at 80–90° C. for fifteen minutes, and then allowed to settle. The time required for the separation of the water from the salt was about fifteen minutes. The water was then poured off and the zinc salt dissolved in twice its volume of benzene. The water and benzene were removed by vacuum distillation at about seven inches vacuum. The maximum temperature of distillation was 100° C. A typical batch in the preparation of the zinc salt of the reaction product of phosphorus pentasulphide and methyl cyclohexanol was as follows:

Grams of salt obtained_________________	980
Per cent yield_______________________	89.5
Time of reaction between NaOH and acid _____________________minutes__	15
Time of reaction between Na salt and $ZnCl_2$ ____________________minutes__	15
Temperature of reaction, °C:	
NaOH+acid______________________	80–90
Na salt+$ZnCl_2$ ___________________	80–90
C. c. of benzol used for dehydration______	2000
Temperature of dehydration °C maximum__	100
Pressure of dehydration_______inches of Hg__	23

Second method

	Grams
Methyl cyclohexanol______________________	865
Phosphorus pentasulphie__________________	333
Zinc oxide_______________________________	100

The methyl cyclohexanol and zinc oxide are placed in a container and heated to 180° F. Then the phosphorus pentasulphide is added in finely divided form so that it will pass a 40 mesh screen. The phosphorus pentasulphide is added over a period of about 40 minutes so that the temperature does not exceed 212° F. The temperature is then raised to 320° F. over a period of one hour and maintained at that temperature for an additional one-half hour. The yield of product is 1167 grams, and may be:

(*a*) dissolved in an equal weight of mineral oil and filtered hot after the addition of 2% by weight of a filter aid such as Filtrol; or
(*b*) dissolved in an equal weight of benzol, filtered, and the benzol removed by evaporation under vacuum.

Third method

The materials and the amounts are the same in this method as in the second method. The methyl cyclohexanol and phosphorus pentasulphide are mixed in a flask and maintained at a temperature of 212° F. by means of a water bath for seven hours. The zinc oxide is then added, and the temperatures raised over a period of one hour to 320° F., and maintained at that temperature for one hour. The yield is 1170 grams, and the final solution and purification may be the same as for the second method.

Fourth method

	Pounds
Methyl cyclohexanol __________________	2975
Phosphorus pentasulphide______________	1137.5
Zinc oxide___________________________	391

The methyl cyclohexanol and phosphorus pentasulphide are heated to 210° F., and the temperature is maintained there for five and one-half hours. The pressure is then reduced to give a vacuum equivalent to five inches of mercury, absolute pressure, for one-half hour to remove the unreacted alcohol, hydrogen sulphide, etc. The mixture is cooled to 140° F. and zinc oxide added. The temperature is maintained at 140° F. for three hours. A vacuum equivalent to three inches of mercury, absolute pressure, is applied and the temperature raised to 210° F., and that temperature maintained for nine hours. Three thousand pounds of a neutral oil are added together with 2% of filter aid, and the solution is filtered at a temperature of about 200° F. The yield of product, exclusive of the oil, is 3800 pounds.

Specific examples of addition agents contemplated for use in this invention are given in Table A below with an indication of the percentages of the various elements found therein by actual analysis.

TABLE A

Example No.	Salt of the reaction product of—	And—	Sulphur	Phosphorus	Metal
			Percent	*Percent*	*Percent*
a	Zinc methyl cyclohexanol	P_2S_5	14.5	7.7	7.5
b	Zinc amyl cyclohexanol	P_2S_5	14.0	7.5	8.5
c	Zinc cosmol	P_2S_5	9.82	4.11	4.36
d	Zinc borneol	P_2S_5	11.0	5.96	6.40
e	Zinc cyclohexanol	P_2S_5	19.3	8.95	8.17

Representative examples of lubricating compositions of the present invention were tested by means of a 2½ H. P. single-cylinder, liquid-cooled, four cycle gasoline engine, rated at 1800 R. P. M. The procedure used was as follows: The engine was loaded by means of a fantype brake which applied a load of 1½ H. P. at 1600 R. P. M. The engine was run for ten minutes at 1200 R. P. M. and then the speed was increased to 1600 R. P. M. The following conditions were maintained constant during the remainder of the test—

Coolant temperature ____°F.. 345
Oil temperature ____°F.. 225
Air fuel ratio ____ 12.7:1

The engine was stopped at 30 hour periods for inspection of piston, piston skirt, piston rings, connecting rod bearings, etc. The engine was equipped with removable connecting-rod bearings consisting of lead-bronze with a steel backing. These bearings were weighed at the beginning of the test and at each 30 hour inspection period. Before each new test, the engine was thoroughly cleaned and equipped with new piston, piston rings and with new connecting rod bearings.

In the following Table B will be found a tabulation of several lubricating compositions of the present invention and in following Table C will be found a tabulation of the characteristics of the lubricant tested, its comparison with an untreated base oil both as to physical properties and performance in the engine tests.

TABLE B

Example	Composition
1	Untreated SAE 30 naphthenic motor oil having a V. I. of 5.
2	Example 1+1% zinc salt of the reaction product of P_2S_5 with methyl cyclohexanol.
3	Example 1+1% zinc salt of the reaction product of P_2S_5 with amyl cyclohexanol.
4	Example 1+2% zinc salt of the reaction product of P_2S_5 with amyl cyclohexanol.
5	Untreated SAE 30 solvent refined Mid-Continent motor oil having a V. I. of 95.
6	Example 5+1% zinc salt of the reaction product of P_2S_5 with methyl cyclohexanol.
7	Example 5+2% zinc salt of the reaction product of P_2S_5 with methyl cyclohexanol.
8	Example 5+2% zinc salt of the reaction product of P_2S_5 with borneol.
9	Untreated conventionally refined SAE 20 Pennsylvania motor oil having a V. I. of 105.
10	Example 9+1% zinc salt of the reaction product of P_2S_5 with methyl cyclohexanol.
11	Example 9+2% zinc salt of the reaction product of P_2S_5 with methyl cyclohexanol.

TABLE C

	1	2	3	4	5	6	7	8	9	10	11
Engine tests at—											
30 hours:											
Piston rating	D	B	A–	A	D	B+	A	B–	D	A–	B+
Number of rings stuck	0	0	0	0	0	0	0	0	0	0	0
Percent filling of oil ring slots	20	0	0	0	0	0	0	0	25	0	0
Bearing loss in mgs.	12	6	10	32	25	16	5	15	8	7	13
60 hours:											
Piston rating	D	C–	B+	A	D	B	A–	C+	D	B+	B
Number of rings stuck	1	0	0	0	0	0	0	0	2	0	0
Percent filling of oil ring slots	90	5	0	0	0	0	0	0	85	0	0
Bearing loss in mgs.	60	12	15	52	69	24	10	14	13	12	23
90 hours:											
Piston rating	----	----	C	A–	D	B–	B+	C–	----	B–	B+
Number of rings stuck	----	----	0	0	1	0	0	0	----	0	0
Percent filling of oil ring slots	----	----	10	0	0	0	0	0	----	0	0
Bearing loss in mgs.	----	----	19	63	107	31	14	16	----	17	30
120 hours:											
Piston rating	----	----	----	A–	----	B	A–	C–	----	C	B
Number of rings stuck	----	----	----	0	----	0	0	0	----	0	0
Percent filling of oil ring slots	----	----	----	0	----	0	0	0	----	5	5
Bearing loss in mgs.	----	----	----	67	----	37	20	24	----	22	38
150 hours:											
Piston rating	----	----	----	A–	----	B–	B+	C	----	----	B
Number of rings stuck	----	----	----	0	----	0	0	0	----	----	0
Percent filling of oil ring slots	----	----	----	0	----	0	0	5	----	----	5
Bearing loss in mgs.	----	----	----	73	----	26	22	27	----	----	38
Drain analysis:											
Percent vis. increase	79.0	82.6	----	74.5	30.2	26.5	28.2	42.0	31.8	56.9	57.5
Conr. carbon residue	2.40	.91	----	1.81	1.43	.73	1.06	1.13	1.39	1.84	2.27
Naphtha insoluble	15.90	33.2	----	27.6	1.2	9.2	1.6	4.2	3.5	1.4	8.6
Acid number	.28	.25	----	.54	1.93	.22	.37	.52	.28	.26	.64
Almen film strength	4	8	----	----	4	10	11	----	4	6	9

The zinc salts produced in accordance with the several modes outlined hereinbefore are all new compositions of matter and are being claimed as such.

One of the principal uses of these zinc salts is in lubricating compositions for the purposes previously explained, and, in the manufacture of a finished composition for use as a lubricant in the crank cases of internal combustion engines, the oil base will be preferably a highly-refined mineral lubricating oil. The range of viscosity for such oil may be from about 100 to about 1000 seconds Saybolt Universal at 100° F. or from about 20 to about 100 seconds Saybolt Universal at 210° F. The preferred range of viscosities for the oil base are from about 150 to about 750 seconds Saybolt Universal at 100° F. or from about 40 to about 75 seconds Saybolt Universal at 210° F.

For certain uses the oil base will be preferably a high V. I. oil. Generally for such uses the oil base should have a V. I. of at least 50 and preferably at least 85 (Dean and Davis System).

The concentration of the zinc salt in the finished lubricant will be largely determined by the particular use for which the composition is designed and the particular characteristics of the base oil or base composition to which they are added. In general, in compounded lubricating oils useful in Diesel engines, the concentration of these zinc salts will range from about .1% to about 4%, and preferably from .5% to 2.5%.

One of the particular uses of the zinc salts of my invention is in lubricating compositions which also contain so-called "detergents," particularly of the metal-containing type. Among the detergents with which these zinc salts may be used in combination are the metal phenates, and especially the alkaline earth metal phenates.

Although the addition agents contemplated hereby have a certain degree of detergent action, particularly in certain types of oils, when used as the sole addition agent, highly advantageous results may be obtained by using these addition agents with others, notably other oil-soluble organic metal compounds which, themselves, effect a higher degree of detergency when used in hydrocarbon oils. Examples of such organic metal compounds are the oil-soluble salts of:

Organic acids produced by oxidation of petroleum fractions
Naphthenic acids
Fatty acids
Halogenated fatty acids, such as dichlor stearic acid
Aromatic acids such as phenyl stearic acid and chlorphenyl stearic acid
Sulphonic acids, e. g. sulphonic acids derived from petroleum
Substituted phosphorus acids containing an organic substituent, e. g. the aliphatic, cycloaliphatic, and aromatic acid esters of phosphoric acid. thio phosphoric acids, phosphorous acid and thio phosphorous acids

The oil-soluble metallic phenates may also be used, such as those derived from such phenolic compounds as:

Alkylated phenols such as cetyl phenol and lauryl phenol
Esters of salicylic acid such as lauryl salicylate and cetyl salicylate
Halogenated alkyl phenols and esters of phenolic acids such as lauryl chlorphenols and lauryl chlorsalicylate
Sulphides and polysulphides derived from phenol and alkylated phenols, such as di(tertiary amyl hydroxy-phenyl) sulphides di(tertiary amyl hydroxy-phenyl) disulphides

Specific examples of such detergent addition agents are the following:

Calcium naphthenate
Cobalt naphthenate
Aluminum naphthenate
Aluminum chlorphenyl stearate
Aluminum cetyl phenate
Calcium oleate
Calcium dichlor stearate
Cobalt dichlor stearate
Calcium phenyl stearate
Calcium chlorphenyl stearate
Calcium cetyl phenate
Calcium lauryl phenate
Magnesium phenate of lauryl salicylate
Calcium lauryl chlorphenate
Magnesium phenate of lauryl chlorsalicylate
Calcium phenate of di(tertiary amyl hydroxy-phenyl) sulphide
Calcium salt of mono-cetyl phosphate
Calcium salt of di-cetyl phosphate
Calcium salt of di-lauryl phosphate
Aluminum salt of mono-cetyl phosphate
Aluminum salt of di-cetyl phosphate
Calcium salt of mahogany acids (petroleum sulphonic acids)
Barium salt of mahogany acids
Sodium salt of mahogany acids
Chromium salt of mahogany acids
Cobalt salt of mahogany acids

The zinc salts of this invention may advantageously be used with detergents comprising combinations of oil-soluble metallic compounds. Examples of such combinations are:

(a) The combination of a metallic phenate with a metallic salt of a substituted phosphorus acid containing an organic substituent.
(b) The combination of an oil-soluble metallic alcoholate with an oil-soluble metallic salt of a substituted phosphorus acid containing an organic substituent.
(c) The combination of an oil-soluble metallic phenate with an oil-soluble metallic salt of a sulphonic acid.
(d) The combination of an oil-soluble metallic alcoholate with an oil-soluble metallic salt of a sulphonic acid.

Specific examples of combinations of this type are:

EXAMPLE I

	Per cent
Calcim cetyl phenate	.5
Calcium cetyl phosphate	.25
Zinc salt of this invention	.5
Mineral lubricating oil	Balance

EXAMPLE II

	Per cent
Calcium cetyl phenate	.5
Calcium cetyl phosphate	.5
Zinc salt of this invention	.75
Mineral lubricating oil	Balance

EXAMPLE III

	Per cent
Aluminum butoxide	.1
Calcium cetyl phosphate	.75
Zinc salt of this invention	.75
Mineral lubricating oil	Balance

EXAMPLE IV

	Per cent
Calcium salt of mahogany acids (sulphonic acid produced by the treatment of a petroleum fraction with fuming sulphuric acid)	1.2
Calcium salt derived from the alkylated phenol sulphide disclosed in U. S. Patent No. 2,139,766	.8
Zinc salt of this invention	.5
Mineral lubricating oil	Balance

EXAMPLE V

	Per cent
Calcium salt of mahogany acids	1.2
Calcium salt of the condensation product of formaldehyde with amyl phenyl	.5
Zinc salt of this invention	.75
Mineral lubricating oil	Balance

EXAMPLE VI

	Per cent
Calcium salt of mahogany acids	1.2
Aluminum butoxide	.1
Zinc salt of this invention	.75
Mineral lubricating oil	Balance

EXAMPLE VII

	Per cent
Calcium salt of an acid produced by the oxidation of a fraction of a solvent-refined paraffinic mineral oil	.7
Calcium salt derived from the alkylated phenol sulphide disclosed in U. S. Patent No. 2,139,766	.15
Zinc salt of this invention	.5
Mineral lubricating oil	Balance

The foregoing specific examples, it will be observed, are in each instance lubricating compositions in which the mineral oil contains the zinc salt of this invention in combination with two other components. These have been given as representative not only of such three-component compositions, but also of two-component compositions which may be produced by the omission, from any of the above-named specific examples, of either one or the other of the two addition agents with which the zinc salt is shown in combination.

Additional specific examples of combinations in which the zinc salt is used with only one other detergent are as follows:

EXAMPLE VIII

	Per cent
Calcium cetyl phenate	.5
Zinc salt of this invention	.5
Mineral lubricating oil	Balance

EXAMPLE IX

	Per cent
Calcium salt of mahogany acids	1.6
Zinc salt of this invention	.75
Mineral lubricating oil	Balance

EXAMPLE X

	Per cent
Magnesium phenate derived from lauryl salicylate	.75
Zinc salt of this invention	2.00
Mineral lubricating oil	Balance

EXAMPLE XI

	Per cent
Calcium phenyl stearate	1.00
Zinc salt of this invention	1.00
Mineral lubricating oil	Balance

EXAMPLE XII

	Per cent
Cobalt chlorphenyl stearate	.5
Zinc salt of this invention	.75
Mineral lubricating oil	Balance

It will be noted that the addition agents of this invention contain, in a single compound, the combination of a cycloaliphatic radicle, a thiophosphate radicle, and the element, zinc. It has been discovered that this combination of constituents produces a material having a particularly desirable combination of properties for use in lubricating oil to be used in the crank cases of internal combustion engines under conditions of severe service. This combination of properties includes the following:

(1) High solubility in oils of high viscosity index;
(2) Detergent action or resistance to sludge-forming tendencies; and
(3) Controlled sulphur activity, contributing corrosion-inhibiting properties and resistance to the formation of acidic materials during oxidation.
(4) Ability to increase film strength of the oil.

In the past, it has not been found possible to compound addition agents which would reduce corrosive tendencies, and the tendency to produce acidic materials during oxidation, without increasing the tendency to form deleterious deposits. By the use of the addition agents of this invention, however, the corrosive tendency and the formation of acidic materials are reduced while, at the same time, reducing the tendency to form harmful deposits.

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims or the equivalent of such be employed.

I, therefore, particularly point out and distinctly claim as my invention:

1. A lubricating composition comprising a major amount of a hydrocarbon oil and a minor amount of the zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with an alkylated cyclohexanol in which substantially all of the alkyl groups have less than six carbon atoms.

2. A lubricating composition comprising a major amount of a hydrocarbon oil and a minor amount of the zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with an alkylated cyclohexanol in which substantially all of the alkyl groups have less than four carbon atoms.

3. A lubricating composition comprising a major amount of mineral lubricating oil and a minor amount of the zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with a methyl-cyclohexanol.

4. A lubricating composition comprising a major amount of mineral lubricating oil and a minor amount of the zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with an amyl-cyclohexanol.

5. A lubricating composition comprising a major amount of mineral lubricating oil and from about .1% to about 4% of the zinc salt of the reaction product obtained by reacting one mole of phosphorus pentasulfide with approximately five moles of methyl-cyclohexanol.

6. A lubricating composition comprising a major proportion of mineral lubricating oil and from about .1% to about 4% of the zinc salt of di(methylcyclohexyl) dithiophosphate.

7. A composition of matter for use as an addition agent in lubricants comprising an oil base and the zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with a methyl-cyclohexanol.

8. A corrosion-inhibiting composition comprising an oil vehicle and in solution therein an effective amount of a product obtained by reacting together zinc oxide, methyl cyclohexanol and phosphorus pentasulfide.

9. A lubricating composition comprising a major proportion of lubricating oil suitable for use in the crank cases of internal combustion engines, with the properties of such composition affecting its use as a lubricant under conditions of extreme stress such as are encountered in Diesel engines and the like, and having its resistance to deterioration and the formation of acidic and varnish forming constituents improved by incorporating therein from about 0.5% to about 2.5% based on the amount of lubricating oil of a zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with an alkylated cyclohexanol in which substantially all of the alkyl groups have less than six carbon atoms.

10. A lubricating composition comprising a major amount of mineral lubricating oil and a minor amount of the zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with a propyl-cyclohexanol.

11. A lubricating composition comprising a major amount of mineral lubricating oil and from about .1% to about 4% of the zinc salt of the reaction product obtained by reacting one mole of phosphorus pentasulfide with approximately five moles of an alkylated cyclohexanol in which substantially all of the alkyl groups have less than six carbon atoms.

12. A lubricating composition comprising a major proportion of mineral lubricating oil and from about .1% to about 4% of the zinc salt of a di(alkylcyclohexyl) dithiophosphate in which substantially all of the alkyl groups have less than six carbon atoms.

13. A composition of matter for use as an addition agent in lubricants comprising an oil base and the zinc salt of an acidic reaction product obtained by reacting phosphorus pentasulfide with an alkylated cyclohexanol in which substantially all of the alkyl groups have less than six carbon atoms.

14. A corrosion-inhibiting composition comprising an oil vehicle and in solution therein an effective amount of a product obtained by reacting together zinc oxide, phosphorus pentasulfide and an alkylated cyclohexanol in which substantially all of the alkyl groups have less than six carbon atoms.

PETER A. ASSEFF.

Patented Mar. 14, 1944 **2,344,392**

UNITED STATES PATENT OFFICE

2,344,392

CRANKCASE LUBRICANT AND CHEMICAL COMPOUND THEREFOR

Elmer W. Cook, New York, N. Y., and William D. Thomas, Jr., Stamford, Conn., assignors to American Cyanamid Company, New York, N. Y., a corporation of Maine

No Drawing. Application November 8, 1941, Serial No. 418,326

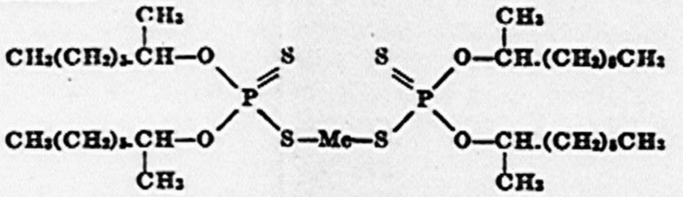
6 Claims. (Cl. 252—48)

This invention relates to a new class of chemical compounds which are useful as addition agents to lubricating oils of the type known as crankcase oil; i. e. hydrocarbon oils of relatively low viscosity and pour point such as are used in the crankcases of internal combustion engines and to lubricating oils of this type containing such additives. More particularly, the invention relates to the heavy metal salts of the dicapryl ester of dithiophosphoric acid and to lubricating oils of the above type containing this class of compounds. By the term "heavy metal salt" in the following specification and claims we mean the salts of the metallic elements of groups II to VIII inclusive of the periodic system and especially such salts as those of the alkaline earth metals magnesium, calcium, strontium and barium and other metals such as zinc, aluminum, chromium, tin, cobalt and nickel which form water-insoluble salts with dicapryl dithiophosphate. Salts of lithium, sodium, potassium, rubidium and cæsium, the alkali metals, are excluded from the invention.

The heavy metal salts of dicapryl dithiophosphoric acid ester are water-insoluble but hydrocarbon oil-soluble compounds. Most of the compounds of this class, including all the alkaline earth metal salts are brownish-yellow solids of low melting point, whereas the zinc salt is a viscous liquid at ordinary temperatures. The compounds as a class are soluble in hydrocarbon oils of the type of 10-W grade lubricating oils to the extent of 30–50% by weight, and are therefore well adapted for manufacture and sale in the form of lubricating oil solutions of fairly high concentration, which can be readily blended with further amounts of oil to concentrations of 0.1–3%, in which they are used.

We have discovered that lubricating oils containing 0.1–3% of heavy metal salts of the dicapryl ester of dithiophosphoric acid possess several remarkable and unexpected properties. We have found that these compounds are detergents for the prevention of sludge deposits in truck, bus, aeroplane and marine gasoline and Diesel engines, and that they also possess extremely important non-oxidizing or antioxidant properties when compared with other metal salt detergents that have previously been used. Moreover, these compounds possess a remarkable degree of stability against decomposition and sludge formation under the conditions of heavy duty use in lubricating oil when it is considered that they are esters of aliphatic secondary alcohols of a type that are known to be considerably less heat-stable than the esters of the corresponding primary alcohols.

Dicapryl dithiophosphoric acid is prepared by reacting capryl alcohol with phosphorous pentasulfide in the ratio of approximately 4 moles of the alcohol for each mole of P_2S_5, the reaction being carried out by heating the two ingredients together at 80–95° C. with stirring for about 2½ hours. When the evolution of H_2S has subsided the resulting dicapryl dithiophosphoric acid ester is dissolved in a mixture of absolute alcohol and benzene and sufficient heavy metal oxide is added to effect neutralization. The mixture is heated to complete the neutralization and a part of the solvent is evaporated to expel the water of neutralization, whereupon the solution is filtered and the filtrate evaporated to recover the product. When an alkaline earth metal oxide is used in this manner the alkaline earth metal salts so obtained have the following probable structural formula



The corresponding salts of metals of higher valency, such as the aluminum and chromium salts have similar structural formulas in which three or more thiophosphoric acid ester radicals are linked through sulfur to the metal.

As is noted above one of the most important features of our invention resides in the addition to lubricating oils of the type of hydrocarbon crankcase oils of the heavy metal salts of the above-defined class, either alone or in admixture with other ingredients, in amounts sufficient to exert sludge-dispersing properties under heavy duty service conditions. The formation of sludge in crankcase oils is intimately related to the production of oxidation products of the type of organic acids and it is another important advantage of the heavy metal dicapryl dithiophosphates that they do not cause an increase in oxygen absorption and consequent sludge formation in the oil. Moreover, our new compounds cooperate effectively with the heavy metal salts of di-(2,4-diamyl phenol) monosulfide described in our Patent No. 2,249,626 and with the alkaline earth metal salts of petroleum sulfonates described in our copending application Serial No. 419,016, filed November 13, 1941, either or both of which may be used to combat oxidation and decomposition in the lubricating oil, and oils containing the heavy metal dicapryl dithiophosphates in admixture with either or both of these compounds constitute another important feature of our invention.

The invention will be further illustrated in detail by the following specific examples, which present results obtained with a representative heavy metal salt of dicapryl dithiophosphate. It should be understood, however, that the invention in its broader aspects is not limited to these examples.

Example 1

260 parts by weight of capryl alcohol

$$[CH_3(CH_2)_5CH(OH)CH_3]$$

were stirred rapidly with 111 parts by weight of finely ground P_2S_5 and heated at 80-95° C. for about 2½ hours. At the end of this time practically all of the P_2S_5 was reacted and the dicapryl dithiophosphoric acid remained as a brown liquid. 354 parts by weight of the product so obtained were dissolved in a mixture of equal parts of absolute ethyl alcohol and benzene and stirred rapidly while 77 parts by weight of powdered barium oxide were added to effect neutralization. The mixture was warmed to complete the neutralization and then heated to boiling and a part of the solvent was evaporated to expel the water of neutralization. The solution was then filtered from any traces of barium salts and the filtrate evaporated. The barium dicapryl dithiophosphate was recovered as a brownish-yellow crystalline material of low melting point, insoluble in water but soluble to the extent of about 50% in 10-W grade Pennsylvania (paraffin base) lubricating oil.

The corresponding zinc salt was prepared by adding 41 parts by weight of finely powdered zinc oxide to a solution of 354 parts of dicapryl dithiophosphoric acid in an alcohol-benzene mixture as described above. The product was obtained as a thin oily liquid which was easily soluble in lubricating oil.

Example 2

The barium dicapryl dithiophosphate was tested in representative Pennsylvania lubricating oils by an accelerated oxidation test known as the catalytic Indiana test. The testing apparatus consists of a constant temperature bath maintained at 341° F. in which a number of large glass test tubes are immersed. 300 cc. samples of the oil under test are poured into these tubes and air at the rate of 10 liters per hour is bubbled through the oil. In order to reproduce the conditions existing in the crankcase of an engine weighed strips of copper-lead alloy are suspended in the oil samples. As metallic surfaces, particularly copper, greatly accelerate the rate of oxidation and decomposition of the oil in the presence of oxygen this is an important factor in the test. Bearing corrosion rates can also be determined by again weighing the strips after 70 hours immersion, which is the usual test period.

The results obtained by the above test procedure are shown in the following table, wherein oil A was a solvent refined 10-W grade oil and oil B was a phenol-extracted 10-W grade oil.

	A. P. I. gravity oxidized oil	Naphtha insoluble	Bearing loss, mg. Cu-Pb	Neut. No.
		Per cent		
Oil A			201	
Oil A+0.6% Ba dicapryl dithiophosphate			3	
Oil B	23.5	17.47	215	4.63
Oil B+0.4% Ba dicapryl dithiophosphate			+22	
Oil B+0.6% Ba dicapryl dithiophosphate	27.3	3.02	+16	3.19

This application is a continuation-in-part of our copending application, Serial No. 401,960, filed July 11, 1941.

What we claim is:

1. A solution of a heavy metal salt of the dicapryl ester of dithiophosphoric acid in lubricating oil.

2. A solution of an alkaline earth metal salt of the dicapryl ester of dithiophosphoric acid in lubricating oil.

3. A solution of the barium salt of the dicapryl ester of dithiophosphoric acid in lubricating oil.

4. A lubricating oil composition comprising a crankcase oil having dissolved therein 0.1-3% of a heavy metal salt of the dicapryl ester of dithiophosphoric acid.

5. A lubricating oil composition comprising a crankcase oil having dissolved therein 0.1-3% of an alkaline earth metal salt of the dicapryl ester of dithiophosphoric acid.

6. A lubricating oil composition comprising a crankcase oil having dissolved therein 0.1-3% of the barium salt of the dicapryl ester of dithiophosphoric acid.

ELMER W. COOK.
WILLIAM D. THOMAS, JR.

Patented July 14, 1942 **2,289,795**

UNITED STATES PATENT OFFICE

2,289,795

LUBRICANT

John G. McNab, Roselle, N. J., assignor to Standard Oil Development Company, a corporation of Delaware

No Drawing. Application September 1, 1939, Serial No. 293,037

10 Claims. (Cl. 252—36)

The present invention relates to improved lubricants and especially to crankcase oils for the lubrication of internal combustion engines, and particularly for high pressure liquid fuel injection engines such as Diesel engines. This application is a continuation in part of application No. 234,899, filed October 14, 1938, now Patent No. 2,233,426, granted March 4, 1941. The invention will be fully understood from the following description.

During the past several years it has been found desirable to add certain polyvalent metal soaps to lubricants, especially such lubricants as are used for automotive engines of the Diesel type. The function of these soaps is complex but the effect is mainly one of maintaining a clean engine condition. The various soaps have peculiarities in behavior depending on the particular metals and to some degree the acid radicals with which they are combined. Certain of the soaps are completely insoluble and others, while soluble, cause geling of the oil. Still others are capable of giving clean engines, but do not appear to be able to reduce engine wear or perhaps even cause excessive engine wear. Thus the various soaps, while their functions may in some cases overlap, are not equivalents in some certain respects.

It has now been found that certain mixed soap compositions are very much superior to compositions containing a single soap. Of the various soaps to be under consideration here, there appear to be three general groups; the first comprises calcium and magnesium soaps which appear to be interchangeable, although they may be used together if desired. The second type of soap is that of aluminum and the third of nickel. The results produced by these various soaps as stated above overlap to some extent, but nevertheless they do not appear to be full equivalents. Improved results are obtained by using a mixture of soaps from any two of these groups, or by using the three types of soap in combination. Thus either a nickel or an aluminum soap may be used along with either calcium or magnesium and the nickel-aluminum combination is also good. Furthermore, improved results have been obtained by use of three soaps, nickel, aluminum and calcium or magnesium.

It has been stated that the type of acids used is important. Any of the fatty acids may be employed but it is preferable to use predominantly saturated fatty acids with melting points below 50° C. The higher melting acids such as stearic and palmitic are less desirable because their soaps have a tendency to be less soluble than those of acids having melting points below 50° C., such as capric, lauric and the like. Various mixed acids may be used such as are obtained from the commercial fats or may be made up synthetically, and it is found that if a substantial portion of unsaturated acids be present, say from about 10 to 30%, oil solubility is more readily obtained. Unsaturated acids such as oleic or erucic can also be used alone. Another type of acids that may be employed is that produced by low temperature oxidation of paraffin wax with air, preferably using catalysts. Highly refined oils may be oxidized in the same way, for example, highly solvent extracted oils or hydrogenated oils, but the most suitable product appears to be the so-called oxidized acids which are obtained from sweater oil. This oil is sweated from petroleum waxes and is oxidized catalytically with air at temperatures from about 110 to 160° C.

Naphthenic acids such as those obtained from Venezuelan, Columbian, Gulf Coast, and Roumanian crudes may also be used in making the soaps.

In manufacturing the soaps, the best procedure is to first obtain a soda or potash soap of the particular organic acid or mixture of acids and then prepare the polyvalent metal soap mixture from the soda or potash soap by double decomposition using water soluble salts of the polyvalent metals, for example chlorides or nitrates. In some instances, the polyvalent soaps may be prepared separately and then mixed together, but it is generally preferable to make a mixture of the polyvalent soaps directly by co-precipitation and this is particularly useful when calcium soaps are employed because they have a tendency to be less soluble than the other metal soaps and the co-precipitation method produces materials which are freely soluble.

The amount of the soap may vary somewhat depending on the particular soaps employed and the service in which they are to be used, but the total amount of the polyvalent metal soap varies in general from about $\frac{1}{10}$ to 3½% of the oil. The various soaps may be used in equal proportions, but good results are obtained over a fairly large range of variation in proportions. It is preferable to provide that each of the two or more soaps is present in not less than $\frac{1}{10}$ of the total soap. Thus if 3% of the total soap is employed, the mixture may contain from about .3 to 2.7% of the aluminum soap with from 2.7 to .3% of calcium, magnesium or nickel soap.

It is found desirable to use soap mixtures which are substantially free from non-saponaceous ingredients such as free acids, esters, lactones, lactides, and the like and this may be accomplished in various ways, but the best method is by extracting the soap mixtures with oxygen-containing solvents such as alcohols, ketones, and the like in which the polyvalent soaps are relatively insoluble.

In spite of what has been said in the paragraph above, it has been found desirable to add small amounts of certain specific acids to the soap mixtures, but these acids are not of the preferred type used in manufacturing polyvalent soap. The best acid to be added appears to be a good grade of stearic acid, but similar high molecular weight saturated acids may be employed instead. This acid is added to the oil or to the soap mixture after extraction and the amount may vary from about .1 to 1% of the total mixture.

The soaps may be added to any type of mineral oil, that is to say lubricating oils of naphthenic, paraffinic or mixed base crudes, but particularly to the distillates of naphthenic crudes and these compositions are especially desirable for the lubrication of Diesel engines. The oils are preferably well refined having viscosities from 45 to 65 seconds Saybolt at 210° F., and it is preferred that they have pour points of 0° F., or below; whether obtained naturally or by dewaxing or by the use of pour depressants is not important. The essential ingredients of the present composition are the two or more polyvalent soaps of the types mentioned above and the hydrocarbon oil, but other ingredients may be employed such as sludge dispersing agents, oxidation inhibitors, thickening agents, sulfur or phosphorus containing compounds, and the like.

EXAMPLE 1

Lubricating oils are made from naphthenic lubricating distillate having a viscosity of about 55 seconds at 210° F. Sample I contained 1% of an aluminum soap of an oxidized sweater oil acid and .5% of free stearic acid. Sample 2 contained 1% of a calcium soap of the same oxidized sweater acids and .5% of naphthenic acid which was used to solubilize the calcium sweater oil acids soap. Sample 3 contained .75% of a mixture of aluminum and calcium sweater oil acids soaps in equal proportions. This mixture was prepared by co-precipitation of the said soaps by addition of aluminum and calcium chlorides to the soda soap of the oxidized sweater oil acids. The blend also contained .5% added free stearic acid.

Each of the three samples described above was subjected to a test over long periods in a Caterpillar single cylinder Diesel engine under comparable running conditions. After each test the pistons were taken down and examined carefully and a demerit rating was assigned to each part depending on its condition, for example, to the rings, slits, and grooves, filter, piston skirt, and liners. In considering these demerit numbers, it should be borne in mind that the lower numbers indicate superior condition. The demerit numbers were then weighted to obtain an overall demerit for the piston as a whole. The amount of oil used was measured as well as the wear on the liner. All of these data are assembled in the following table:

Caterpillar single cylinder engine tests

Eng. No.	Run No.	Hours	Overall piston demerit	Ring edge sharp	Demerits							Oil cons. BHP hrs./gallon	Liner wear per 1,000 hrs.	
					Ring zone cleanliness			Carbon	Skirt and liner	Varnish on skirt	Oil filter			
					Slits	Grooves	SGLS*						Max.	Av.
					SAMPLE 1									
C-2	3	500	1.35	0.17	0.0	3.1	1.72	4.6	0.92	1.0	2	3,290	0.0046	0.0015
	--------	1,000	2.00	0.67	0.5	4.5	2.52	5.0	1.92	0.8	5	3,580	0.0045	0.0016
					SAMPLE 2									
C-1	17	500	2.25	0.17	2.0	4.2	2.03	4.8	2.33	4	4	3,480	0.0028	0.0012
					SAMPLE 3									
C-2	8	500	1.12	0.25	0.0	2.3	1.28	4.4	0.67	0.25	1.0	2,770	0.0036	0.0016
	--------	1,000	1.45	1.00	0.25	2.6	2.01	4.3	0.67	0.00	2.0	3,545	0.0022	0.0009

*Cleanliness of ring slits, grooves, lands, and sides.

From the above data it is clear that the oil containing both the calcium and the aluminum soap, Sample 3, is much superior to either of the blends in which the single soaps are used. The overall demerit rating is much better and only in one comparison is it inferior to either of the other samples. This is in respect to the sharpness of the ring edge, but it is clear that this is not of very great importance and is much overshadowed by the improved condition of the slits, grooves, etc., the decrease in the varnish and the improved filter condition.

It has now also been found that in addition to the metal soaps of natural and synthetic fatty acids and naphthenic acid as disclosed in the parent application referred to, it is possible to use a large number of other polyvalent, oil-soluble metal salts. Broadly these metal compounds may be considered as having the general formula

$$M(XY)_n$$

where M is a metal in one of the two groups of the alkaline earth metals and aluminum; X is either oxygen or sulfur, and Y is an organic radical, the letter n representing the valency of the metal M. Some of the classes of metal salts which come within the general scope of this invention but were not disclosed in the parent application are the phenolates, thio-phenolates, as well as alkylated derivatives of either of them, sulfide derivatives of phenolates, thio-phenolates and alkylated derivatives thereof; alcoholates, carbamates and thio carbamates, xanthates and enolates. In the case of salts of the alkyl phenol sulfides, the metal M in the above formula may also represent nickel. It is particularly preferred to use alkylated and/or sulfide derivatives of the phenolates and thio-phenolates because these compounds not only posses superior oil-solubility but also are less corrosive to bearings of the lead, bronze and cadmium-silver type. In addition they frequently impart superior lubricant action and give lower demerit ratings in engine tests.

It has also been found that the salts of varium may be properly included along with calcium and magnesium salts in the general field of alkaline earth metal salts as far as this invention is concerned.

It has been found that the metal compounds of this invention are particularly applicable in Diesel lubricants comprising a major proportion of a refined mineral oil having a viscosity of 40 to 75 seconds Saybolt at 210° F. and preferably having a pour point of 30° F. or lower.

By the use of the metal salts according to this invention such small amounts of metal compounds are used, and particularly when used in a mineral oil of a sufficiently low viscosity and low pour point to be suitable for a Diesel lubricant, that no substantial thickening is effected by the incorporation of the metal salt. For instance, the increase in viscosity is in most cases less than 10% of the Saybolt viscosity at 210° F.

Although as stated previously the total amount of metal compounds to be used should be between the approximate limits of 0.1 to 3.5% by weight of the mineral oil used, it is normally preferred to maintain the total amount of metal compounds added between the approximate limits of 0.2 and 1.5%. Larger quantities of the additive usually fail to confer enough additional benefit to justify the additional cost. Also although it is desired that in a mixture of the several different types of metal compounds, each type should be present in an amount corresponding to at least 0.1 of the total amount of the metal compounds present, it is ordinarily preferred for practical operation that each type of metal compound be present in at least ¼ the amount of the total compound used. Equal proportions or a ratio of 1 to 2 are in most cases satisfactory for practical operation.

For the sake of illustration, a number of specific examples of the metal compound falling within the scope of this invention are listed herewith. (Under the first group listed, namely the phenolates, corresponding compounds are given for all of the four different metals, namely calcium, magnesium, barium and aluminum, but in the rest of the classes, to avoid unnecessary permutations, only the calcium compound is given as representative of the various metals.)

Phenolates

Calcium phenolate, $Ca(OC_6H_5)_2$
Magnesium phenolate, $Mg(OC_6H_5)_2$
Barium phenolate, $Ba(OC_6H_5)_2$
Aluminum phenolate, $Al(OC_6H_5)_3$

Thio-phenolates

Calcium thio-phenolate, $Ca(SC_6H_5)_2$

Mixed oxy- and thio-phenolates

Calcium oxy-thio-phenolate, $Ca(OC_6H_5)SC_6H_5$

Alkylated phenolates

Calcium di-tertiary butyl phenolate, $Ca(OC_6H_4 \cdot t\text{-}C_8H_{17})_2$

Alkylated thio-phenolates

Calcium tertiary amyl thio-phenolate, $Ca(SC_6H_4 \cdot t\text{-}C_5H_{11})_2$

Sulfide phenolates

Calcium phenolate sulfide, $Ca(O \cdot C_6H_4)_2S$

Alkylated phenolate sulfides

Calcium tertiary amyl phenolate sulfide, $Ca(O \cdot C_6H_3 \cdot t\text{-}C_5H_{11})_2S$
Calcium tertiary amyl phenolate disulfide, $Ca(O \cdot C_6H_3 \cdot t\text{-}C_5H_{11})_2S_2$

Thio-phenolate sulfides

Calcium thio-phenolate sulfide, $Ca(S \cdot C_6H_4)_2S$

Alkylated thio-phenolate sulfides

Calcium tertiary amyl thio-phenolate sulfide, $Ca[S \cdot C_6H_3(t\text{-}C_5H_{11})]_2S$

Alcoholates

Calcium methylate, $Ca(O \cdot CH_3)_2$
Calcium ethylate, $Ca(O \cdot C_2H_5)_2$
Calcium propylate, $Ca(O \cdot C_3H_7)_2$
Calcium isopropylate, $Ca(O \cdot i\text{-}C_3H_7)_2$
Calcium hexylate, $Ca(O \cdot C_6H_{13})_2$
Calcium octylate, $Ca(O \cdot C_8H_{17})_2$
Calcium octadecylate, $Ca(O \cdot C_{18}H_{37})_2$
Calcium methylate octadecylate, $Ca(O \cdot CH_3)O \cdot C_{18}H_{13}$

Thio alcoholates

Calcium thio-hexylate, $Ca(S \cdot C_6H_{13})_2$

Carbamates

Calcium carbamates of the general formula $Ca(OOC \cdot NR_2)_2$, in which R is a hydrocarbon radical preferably an alkyl group having at least 4 carbon atoms such as butyl or octyl, or a cyclic hydrocarbon group such as cycloalkyl, aryl, etc.

Thio-carbamates

Calcium di-butyl mono-thio carbamates, $Ca[S \cdot OC \cdot N(C_4H_9)_2]_2$

Di-thio carbamates

Calcium di-butyl di-thio carbamate, $Ca[S \cdot SC \cdot N(C_4H_9)_2]_2$

Xanthates

Calcium amyl xanthate, $Ca(S \cdot CS \cdot OC_5H_{11})_2$

Thio-xanthates

Calcium amyl thio-xanthate, $Ca(S \cdot CS \cdot SC_5H_{11})_2$

Phosphites

Calcium di-octyl phosphite, $Ca[OP \cdot (OC_8H_{17})_2]_2$

Thio-phosphites

Calcium di-octyl thio-phosphite, $Ca[SP \cdot (OC_8H_{17})_2]_2$

Phosphates

Calcium di-octyl phosphate, $Ca[OP \cdot O \cdot (OC_8H_{17})_2]_2$

Thio-phosphates

Calcium di-octyl di-thio-phosphate, $Ca[SP \cdot S \cdot (OC_8H_{17})_2]_2$

Enolates

Calcium enolate hexyl ester of aceto-acetic acid, $Ca[OC(CH_3){=}CH\text{-}CO \cdot O \cdot C_6H_{13}]_2$

Many of the compounds listed are only sparingly oil soluble and can consequently only be employed with the assistance of a mutual solvent such as, for instance, stearic acid, stearyl alcohol, dibutyl phthalate, tricresyl phosphate, or other high boiling compound. It is therefore generally preferred so to alter their structure by alkylation as to obtain sufficient inherent oil solubility to render the use of mutual solvents unnecessary. Where compounds contain a sulfur group it is usually sufficient to have the equivalent of two alkyl chains of at least 4 to 5 carbon atoms present as exemplified by:

Calcium tertiary amyl phenolate sulfide,
$Ca(O \cdot C_6H_3 \cdot tC_5H_{11})_2S$

When sulfur groups are absent even more carbons in alkyl chains are required and the equivalent of two alkyl chains of at least 8 carbons appears essential, as in:

Calcium octyl phenolate, $Ca(OC_6H_4 \cdot C_8H_{17})_2$
Calcium di-butyl phosphite, $Ca(OP \cdot (C_4H_9)_2)_2$

It is obvious that as the solvent power of an oil varies with its crude source the exact minimum requirements, as given above will also vary.

In the various compounds discussed above, which involve sulfur as one constituent, it is understood that the corresponding selenium or tellurium compounds could also be used.

For further illustration the following additional examples are given of lubricants prepared according to this invention and comprising combinations of various metal salts.

EXAMPLE 2

To the same mineral oil described in Example 1, metallic salts of tertiary amyl phenol sulfide were added. Sample 1 contained 1% of the nickel salt, sample 2 contained 1% of the barium salt, and sample 3 contained 0.5% of each.

Samples 1 and 2 were run in a single cylinder Caterpillar Diesel engine for 60 hours under comparable operating conditions. The same sample 2, and sample 3, were run in a second similar single cylinder Caterpillar Diesel engine, also under comparable operating conditions, though not identical with those employed in the first engine.

After each test the engine was dismantled, examined and rated following the procedure described in Example 1. The results are assembled in the following table:

Caterpillar single cylinder engine tests

Engine No.	Run No.	Sample No.	Metal used	Rings stuck	Engine demerits				
					Heat groove	Ring grooves and sides		Piston skirt varnish	Oil filter
						#1 and #2	#3 and #4		
1	35J	1	1.0% nickel salt	0	7.0	12.5	15.0	0.75	3.0
1	35K	2	1.0% barium salt	0	2.0	16.0	13.0	0.75	2.0
2	19K	2	1.0% barium salt	1	7.5	11.5	28.5	1.25	2.0
2	19J	3	0.5% nickel salt+0.5% barium salt	0	6.0	9.0	16.0	1.0	1.5

From the above data it is apparent that in No. 1 engine the oil containing 1.0% of the barium salt (sample 2) performed better than that with 1.0% of the nickel salt (sample 1), and that the oil blended with the mixture of 0.5% of each (sample 3) performed better than the 1.0% barium salt composition (sample 2). It may be concluded therefore that the use of the two additives in combination give results superior to those obtainable with either alone.

EXAMPLE 3

To the above uncompounded mineral oil 0.5% of the nickel salt of tertiary amyl phenol sulfide and 0.5% of the calcium salt of the enol form of the hexyl ester of aceto acetic acid was added. The resulting blend was a clear, fluid oil not appreciably different from the original mineral oil in appearance. When mixed with carbon black, the latter settled only very slowly indicating that the composition possessed dispersing ability not characteristic of the mineral oil alone.

It has been pointed out above that many of these compounds, particularly those containing sulfur, are non-corrosive to such bearing metals as the lead bronzes and the cadmium-silver alloys. Others, however, lack this desirable property and are therefore used most advantageously in conjunction with such known corrosion preventions as sulfur compounds and sulfurized oils, anti-oxidants (particularly phenols) metal deactivators (particularly organic phosphites), etc. For greater oiliness and lubricity, oiliness agents may be included; thickeners, viscosity-index improving agents, pour depressors and dyes may all be present in the finished lubricating composition.

This invention is not to be limited by any specific examples which have been presented herein solely for the purpose of illustration, but only by the following claims, in which it is desired to claim all novelty inherent in the invention.

I claim:

1. An improved lubricating composition comprising a mineral lubricating oil and small amounts of polyvalent metal salts selected from each of the groups consisting of aluminum, and alkaline earth metal salts, the total amounts of such metal salts amounting to from 0.1% to 3.5% by weight of the oil and the amount of each type of metal salt being at least one-tenth of the total metal salt present.

2. Lubricant according to claim 1 in which the various metal salts used have the general formula

$$M(XY)_n$$

in which M represents the metal, X represents oxygen, sulfur, selenium or tellurium, and Y represents an organic radical, the subscript n representing a numeral corresponding to valence of metal M.

3. Lubricant according to claim 1 in which the metal salts used are compounds containing an organic radical selected from the group consisting of alkyl, aryl, alkylated aryl, carbamyl, xan-

thyl, thio-xanthyl, enolyl, acyl and thio-acyl, phosphyl and thio-phosphyl.

4. Lubricant according to claim 1 in which at least one of the metal compounds has the formula

$$M(O \cdot C_6H_4R)_x$$

in which M is the metal and R is an alkyl group sufficiently large to make the compound soluble in mineral oil.

5. An improved lubricant for Diesel engines comprising a major proportion of a refined mineral oil having a viscosity of about 40 to 75 seconds Saybolt at 210° F., and a pour point at least as low as 0° F. and containing 0.1 to 3.5% of polyvalent metal salts, at least ¼ of the total amount of metal salts present being selected from each of the groups consisting of aluminum, and alkaline earth metal salt.

6. An improved lubricant for Diesel engines comprising essentially mineral oil having a viscosity of 40 to 75 seconds Saybolt at 210° F., and a pour point at least as low as 30° F. into which has been dissolved from 0.1 to 3.5% by weight of oil-soluble polyvalent metal salts having the general formula

$$M(O \cdot C_6H_3R)_2S$$

in which M is the metal and R is an oil-solubility-imparting alkyl group, said metal salts being selected from at least two of the groups consisting of aluminum, nickel and alkaline earth metal salts, the amount of each type of salt present being at least ¼ of the total amount of metal salt present.

7. A lubricating composition comprising a major proportion of a mineral lubricating oil and a small amount each of a nickel alkyl phenolate sulfide and barium alkyl phenolate sulfide.

8. Lubricant according to claim 6 in which the said lubricant contains one of said salts of aluminum and one of said salts of an alkaline earth metal.

9. Lubricant according to claim 6 in which the said lubricant contains one of said salts of nickel and one of said salts of an alkaline earth metal.

10. An improved lubricating composition comprising a mineral lubricating oil and small amounts of polyvalent metal salts selected from at least two of the groups consisting of aluminum, nickel and alkaline earth metal salts, the said nickel salt being, in each instance in which it is used, a compound having the general formula Ni $(OC_6H_3R)_2S$, in which R is an oil-solubility-imparting alkyl group, the total amounts of such metal salts amounting to from 0.1% to 3.5% by weight of the oil and the amount of each type of metal salt being at least one-tenth of the total metal salt present.

JOHN G. McNAB.

Patented Dec. 8, 1936 **2,063,629**

UNITED STATES PATENT OFFICE

2,063,629

ESTERS OF THE THIO ACIDS OF PHOSPHORUS

Paul L. Salzberg, Wilmington, and James H. Werntz, Marshallton, Del., assignors to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

No Drawing. Application February 19, 1935, Serial No. 7,216

22 Claims. (Cl. 260—99.20)

This invention relates to new esters of the thio acids of phosphorus and more particularly to the long chain aliphatic esters of such acids.

The aliphatic esters of orthophosphoric acids are known. The orthophosphoric esters of the low molecular weight alcohols have not proved to be entirely satisfactory for commercial use for the reason that they do not have the marked surface active effect desired in compounds of this type in the uses for which they are intended to be employed. The orthophosphoric esters of the longer chain aliphatic alcohols have the desired surface active effect but are generally solids at ordinary room temperatures and are insufficiently soluble in oils and hydrocarbon solvents to enable full advantage to be taken of their desirable properties.

An object of the present invention is to provide new chemical compounds having the advantageous features of prior compounds but without all of their disadvantages. A further object of this invention is to provide long chain aliphatic esters of the thio acids of phosphorus which are new chemical compounds. A still further object is to provide methods for preparing such new chemical compounds. Other objects are to provide new compositions of matter and to advance the art. Still other objects will appear hereinafter.

These objects may be accomplished in accordance with our invention which comprises reacting an esterifying derivative of a thio acid of phosphorus with at least one molecular proportion of an aliphatic alcohol containing at least ten carbon atoms or reacting an esterifying derivative of an acid of phosphorus with at least one molecular proportion of an aliphatic mercaptan containing at least ten carbon atoms whereby aliphatic esters of the thio acids of phosphorus are obtained. These compounds are new chemical compounds not known heretofore and have many advantageous properties over phosphoric esters heretofore known.

By the term "a thio acid of phosphorus" we intend to include the mono, di, tri and tetra thio ortho-phosphoric acids, the thio-meta-phosphoric acids, the thio-pyrophosphoric acids, the thio-hypophosphoric acids and also the thio phosphorus acids including the ortho, pyro and hypo acids. However, the thiophosphoric acids are generally the cheapest to manufacture and have the most desirable properties and of these the thio ortho-phosphoric acids are preferred both for ease and cheapness of manufacture and for their unusually desirable properties.

The esters of our invention contain at least one aliphatic chain of at least ten carbon atoms. They may contain 2, 3 or 4 of such chains. They may contain aliphatic chains of a lesser number of carbon atoms. These aliphatic chains may be derived from either saturated or unsaturated aliphatic alcohols and are preferably hydrocarbon chains. They may also contain phenolic radicals. In other words, the esters of our invention may be either neutral or acid esters and may be homogeneous or mixed esters.

The esters of dithio ortho-phosphoric acid may be prepared by reacting at least one molecular proportion of the desired alcohol with a mixture of phosphorus and sulphur or with P_2S_5. For example, two molecular proportions of dodecyl alcohol, when reacted with P_2S_5, forms didodecyl dithiophosphate. An inert organic solvent such as benzene or toluene may be employed to better control the reaction. The reaction will generally be carried out under a reflux condenser at refluxing temperatures and under a slight vacuum in order to remove hydrogen sulfide which is formed. If desired, the alcohol may be first reacted with sodium to form the corresponding alcoholate before reacting with the phosphorus pentasulfide.

The monothio ortho-phosphates may be prepared by reacting one or more molecular proportions of the alcohol or alcoholate with phosphorus sulfochloride $PSCl_3$. For example, when three molecular proportions of sodium dodecyloxide were reacted with $PSCl_3$, tridodecyl monothio ortho-phosphate was obtained. When one molecular proportion of decyl alcohol was reacted with $PSCl_3$, the mono decyl monothio phosphate was obtained.

Still other esters of thio ortho-phosphoric acids may be prepared by reacting mercaptans such as tetradecyl mercaptan with phosphorus oxychloride $POCl_3$. The compounds produced by this method have each alkyl chain connected to the phosphorus through a sulphur atom. The resulting product will be a mono, di or tri-thio phosphate, depending upon the molecular proportions of the mercaptan to the phosphorus oxychloride. If the phosphorus oxychloride is substituted by phosphorus penta sulphide the tetra thio phosphate will be produced. A dithio phosphate produced by this method will be isomeric with that obtained by reacting the alcohol with phosphorus pentasulfide. It will be apparent that a variety of esters of the thio acids of phosphorus may be obtained by employing other esterifying derivatives of the acids of phosphorus in place of the phosphorus oxychloride, phosphorus pentasulfide, phosphorus sulfochloride, etc.

In order to more clearly illustrate our invention and the preferred modes of carrying the same into effect, the following examples are given:

Example 1

Twenty-two and two-tenths parts by weight of phosphorus pentasulfide were suspended in 258 parts by weight of toluene. To this, 74.4 parts by weight of dodecyl alcohol were added slowly. The mixture was heated at 725 mm. under a reflux condenser for 18 hours. The resulting reaction product forms stable emulsions when mixed with water.

This reaction product was treated with the calculated amount of aniline and heated under a vacuum to remove most of the toluene. The thick syrup which remained crystallized when chilled in ice water. The salt was recrystallized from toluene in which it is soluble at room temperatures. The white crystals were then dissolved in ether and aniline hydrochloride was precipitated by passing in dry hydrogen chloride. The aniline hydrochloride was filtered off and the filtrate evaporated. The oily residue became solid when chilled to about —50° C. but melted to an oil when warmed up to 20° C. It did not crystallize in an ice-salt mixture. The oil was heated under a vacuum to remove residual solvents. This oil analyzed 6.3% of phosphorus. (Theory for didodecyl dithiophosphate is 6.6% phosphorus.) The didodecyl dithio phosphate when added to water was sufficiently soluble to render the water acid in reaction.

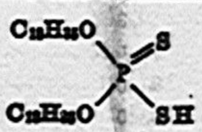
The p-nitroaniline salt of the dithiophosphate was prepared by dissolving it in toluene and treating with p-nitroaniline. This salt was quite soluble in toluene and alcohol at ordinary temperatures.

The pyridine salt was prepared by adding the calculated amount of pyridine to a water suspension of the didodecyl dithio phosphate and the solution shaken. The resulting solution foamed and exhibited a cleansing action when applied to the skin.

The sodium salt of didodecyl dithio phosphate, made by treating didodecyl dithiophosphate with an aqueous solution of sodium carbonate, is not soluble in water.

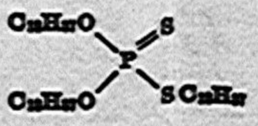
Example 2

When four molecular proportions of 9,10-octadecenyl alcohol were reacted with one molecular proportion of phosphorus pentasulfide in accordance with the procedure of Example 1, dioctadecenyl dithio ortho-phosphate was produced. This compound has the formula



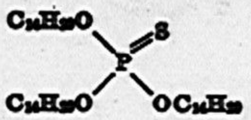
Example 3

When five molecular proportions of octadecyl alcohol were reacted with one molecular proportion of phosphorus pentasulfide, trioctadecyl dithio ortho-phosphate was obtained. This compound has the following formula



Example 4

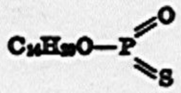
When three molecular proportions of tetradecyl alcohol were reacted with one molecular proportion of phosphorus sulphur chloride, tritetradecyl monothio ortho-phosphate having the formula



was obtained.

Example 5

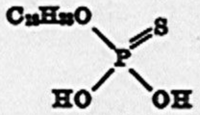
Monotetradecyl thiometaphosphate having the formula



was obtained by treating tritetradecyl monothio ortho-phosphate with concentrated sulphuric acid.

Example 6

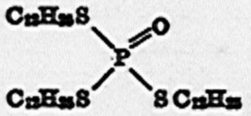
Monocetyl monothio o-phosphate having the formula



was prepared by reacting one mol. of cetyl alcohol with one mol. of $PSCl_3$ in accordance with the procedure of Example 4, followed by hydrolysis.

Example 7

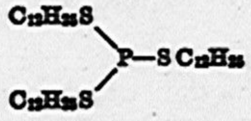
Tridodecyl trithio ortho-phosphate having the formula



may be made by reacting three mols of dodecyl mercaptan with one mol. of phosphorus oxychloride.

Example 8

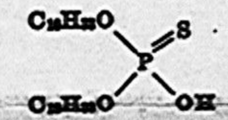
Tridodecyl trithio phosphate



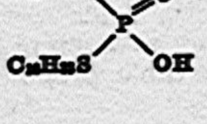
may be obtained by reacting one mol. P_2S_3 with three mols of dodecyl alcohol. This compound is preferably obtained by reacting three mols of dodecyl mercaptan with phosphorus trichloride PCl_3.

Example 9

The 0,0-dicetyl monothio ortho-phosphate

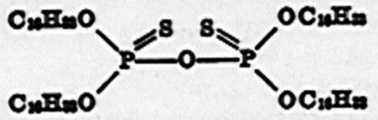


may be obtained by reacting cetyl alcohol with phosphorus penta-sulfide in accordance with the procedure of Example 1. The resulting product apparently contains a substantial amount of O,S-dicetyl monothio orthophosphate having the formula



Example 10

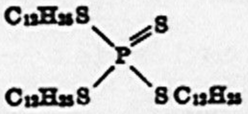
The tetracetyl dithiopyrophosphate



may be obtained by treating tricetyl monothiophosphate with concentrated sulphuric acid.

Example 11

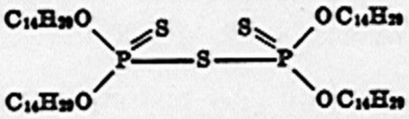
Tridodecyl tetra thio o-phosphate having the formula



may be obtained by reacting three mols of dodecyl mercaptan with one mol. of phosphorus pentasulfide.

Example 12

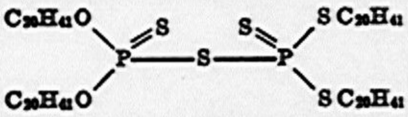
Tetratetradecyl trithiopyrophosphate



may be obtained by reacting four mols of tetradecyl alcohol with one mol. of phosphorus trisulfotetrabromide $P_2S_3Br_4$.

Example 13

Tetraeicosyl penta-thiopyrophosphate



may be obtained by reacting two mols of eicosyl alcohol and two mols of eicosyl mercaptan with one mol. of phosphorus trisulfotetrabromide.

Example 14

One gram molecular weight of phosphorus pentasulfide was suspended in 2580 parts by weight of toluene. To this four gram molecular weights of the mixture of alcohols obtained by the carboxylic reduction of coconut oil were added slowly. The mixture was heated at 725 mm. at the reflux temperature under a reflux condenser for 18 hours. The resulting mixture of esters and mixed esters of dithio phosphoric acid was an oil which formed stable emulsions when mixed with water. This oil blends readily with lubricating oil and in this respect offers an advantage over a pure didodecyl dithio phosphate which blends less readily with lubricating oil.

The above examples are merely illustrative of some of the compounds embraced within our invention and the preferred modes of preparing such compounds. Other similar compounds may be prepared from other alcohols such as decyl, undecenyl, hexacosyl, eleostearyl, linoleyl, ricinoleyl, abietyl and the like alcohols and mercaptans. Also, mixtures of esters containing mixed esters may be prepared by reacting a mixture of such alcohols with an esterifying derivative of a thio acid of phosphorus or a mixture of the corresponding mercaptans with an esterifying derivative of an acid of phosphorus. When such mixtures are employed, it will, in general, be desirable to employ such a mixture obtainable from the alcohols obtained by the carboxylic reduction of a natural fatty oil such as sperm oil and coconut oil or the acids present therein. Other mixtures of esters comprising mixed esters may be obtainable by reacting an esterifying derivative of a thioacid of phosphorus with such an alcohol or mixture of alcohols admixed with other alcohols or phenols. Our invention also includes esters prepared from secondary alcohols derived from the olefins obtained by cracking paraffin wax hydrocarbons.

If pure mixed esters are desired, they may be obtained by reacting one or two mols of an alcohol or mixture of alcohols containing at least ten carbon atoms with the esterifying derivative of a thio acid of phosphorus or their corresponding mercaptans with an esterifying derivative of an acid of phosphorus and then reacting the resulting product with one or more mols of a different alcohol, a phenol or mercaptan.

Our invention embraces the acid esters and their salts such as the alkali, alkaline earth, calcium, zinc, lead or ammonium salts. By "ammonium salts", we intend to include the salts of amines such as, for example, monododecyl amine, diethyl amine, cyclohexylamine, triethanolamine, aniline, nitroaniline, pyridine, naphthylamine, o-toluidine, diphenyl guanidine and the like as well as the salts of ammonia.

In the above examples, we have disclosed toluene as a diluent for the reaction. We may use other inert solvents or no solvents at all. We prefer to use a solvent which is effective for the solution of both the alcohol and the resulting thiophosphate but which is a non-solvent for impurities in the reagents. For example, such solvents as chlorobenzene, carbon tetrachloride, benzene and the like or mixtures of such solvents such as a mixture of carbon tetrachloride and toluene will be found to be satisfactory.

The compounds of our invention may be employed as flotation agents for the separation of various types of minerals, or as parasiticides or as acid inhibitors in metal pickling baths or as corrosion inhibitors to retard the attack of metals by oxygen in aqueous solutions or in lubricants for bearings which operate under high pressures or as accelerators for the vulcanization of rubber. Our compounds will have advantages over similar compounds for these purposes. For example, didodecyl dithiophosphate has the advantage over diamyl dithiophosphate in that the aniline salt of didodecyl dithiophosphate is more soluble in hydrocarbon solvents than the aniline salt of diamyl dithiophosphate. Also, the sodium salt of didodecyl is substantially insoluble in water, whereas the sodium salt of diamyldithiophosphate is readily soluble in water. The dithiophosphates are generally oils at ordinary temperatures, whereas, the corresponding phosphates, such as didodecyl o-phosphate, are solids. This is an advantage for many technical applications such as their use as flotation agents, in pharmaceuticals and as acid corrosion inhibitors. Furthermore, the sulphur content of our compounds renders them more effective for many technical applications than the corresponding phosphates containing no sulphur; for example, when employed as parasiticides, flotation agents, or as acid inhibitors. Furthermore, our compounds are more desirable as assistants for use in extreme pressure lubricants over the corresponding phosphates containing no sulphur and the lower molecular weight dithiophosphates because of their great solubility in the oils and their higher degree of oiliness.

While we have disclosed the preferred embodiments of our invention, it will be readily apparent that many variations and modifications may be made therein without departing from the spirit of our invention. Accordingly, the scope of our invention is to be limited solely by the appended claims construed as broadly as is permissible in view of the prior art.

We claim:

1. An aliphatic ester of a thio-acid of phosphorus in which at least one aliphatic radical contains at least 10 carbon atoms.

2. An aliphatic ester of a thio phosphoric acid in which at least one aliphatic radical contains at least 10 carbon atoms.

3. An aliphatic ester of a thio o-phosphoric acid in which at least one aliphatic radical contains at least 10 carbon atoms.

4. An alkyl ester of a thio-acid of phosphorus in which at least one alkyl radical contains at least 10 carbon atoms.

5. An alkyl ester of a thio phosphoric acid in which at least one alkyl radical contains at least 10 carbon atoms.

6. An alkyl ester of a thio o-phosphoric acid in which at least one alkyl radical contains at least 10 carbon atoms.

7. An aliphatic ester of a dithio-acid of phosphorus in which at least one aliphatic radical contains at least 10 carbon atoms.

8. An aliphatic ester of a dithio phosphoric acid in which at least one aliphatic radical contains at least 10 carbon atoms.

9. An aliphatic ester of dithio orthophosphoric acid in which at least one aliphatic radical contains at least 10 carbon atoms.

10. A dialiphatic ester of a thio-acid of phosphorus, each aliphatic group containing at least 10 carbon atoms.

11. A dialiphatic ester of a thio o-phosphoric acid, each aliphatic group containing at least 10 carbon atoms.

12. A dialiphatic ester of a dithio-acid of phosphorus, each aliphatic group containing at least 10 carbon atoms.

13. A dialiphatic ester of dithio o-phosphoric acid, each aliphatic group containing at least 10 carbon atoms.

14. A dodecyl ester of a thio-acid of phosphorus.

15. A dodecyl ester of a thio o-phosphoric acid.

16. A dodecyl ester of a dithio-acid of phosphorus.

17. A dodecyl ester of dithio o-phosphoric acid.

18. A didodecyl ester of a thio-acid of phosphorus.

19. A didodecyl ester of a thio o-phosphoric acid.

20. A didodecyl ester of a dithio acid of phosphorus.

21. Didodecyl dithio o-phosphoric acid.

22. Di-(9,10-octadecenyl) dithio ortho-phosphate.

PAUL L. SALZBERG.
JAMES H. WERNTZ.

Patented Apr. 21, 1942 **2,280,419**

UNITED STATES PATENT OFFICE

2,280,419

COMPOUNDED OIL

Chester E. Wilson, San Pedro, Calif.

No Drawing. Application July 16, 1940,
Serial No. 345,844

24 Claims. (Cl. 252—33)

This invention relates to mineral lubricating oils which have been modified by the addition of constituents to impart to them special characteristics especially adapting them to severe service uses, such as are encountered in Diesel engines, high output aviation engines and the like.

In Diesel and similar engines the high temperatures developed in the cylinders tend to act upon lubricating oils to cause the deposition of resinous and varnish-like products on the pistons and elsewhere to produce lacquer-like coatings and carbonaceous materials which tend to cause ring and valve sticking and interfere with engine operation. Furthermore, fuel residues from incomplete combustion of fuel contribute to the deposition of lacquer-like and carbonaceous materials in the engine. Many oils used for these purposes also develop corrosive tendencies toward highly corrosion-sensitive bearings.

Primarily, the object of this invention is to produce for such severe service conditions as are encountered in Diesel engines, lubricating oils which possess detergent properties and thereby avoid the deposition of lacquer and varnish-like materials upon pistons and overcome carbon deposition behind the rings, and which will also be non-corrosive to highly corrosion-sensitive bearings of the copper-lead and cadmium-silver type. By detergent property is meant that property of preventing the deposition of such materials or that property of lifting or removing such materials, if it be that they are actually deposited, whereby, whatever the action, the sticking of valves and rings which has been common ordinarily in the lubrication of severe service engines, such as Diesel engines, is overcome or prevented.

The present invention resides in mineral lubricating oils containing small amounts in the order of about 0.3% to 2.0% or 3.0% of an oil-soluble metal salt of a sulfonic acid to insure good detergency, together with a quantity of the same order of oil-soluble metal salt of high molecular weight non-carboxylic weak acid having an ionization constant below about 5×10^{-6}. Such acids are the phenols, thiophenols, alcohols, enols, oximes and sulfonamides to assist detergency and avoid development of the mentioned corrosive conditions. The description of the sulfonic acids and the weak acids are detailed hereinafter. The metal sulfonate and the metal salts of the above mentioned weak acids when present together in a mineral lubricating oil in the amounts indicated above produce an oil which is non-corrosive to alloy bearings, such as those composed of cadmium and silver or copper and lead. Furthermore, this oil has a reduced tendency toward deposition of varnish-like, resinous or carbonaceous materials in the combustion cylinders or on the pistons or piston rings or the like.

As stated above, my new composition contains a small quantity of an oil-soluble metal salt of sulfonic acids, such as the so-called "mahogany" acids. The sulfonate is preferably in the form of the calcium salt. However, the other alkaline earth metals, such as barium, strontium and magnesium may be employed. The salts of the weak non-carboxylic acids are from acids having ionization constants of 5×10^{-6} or less and preferably 1×10^{-6} or less, such as 1×10^{-8} or within the range of 1×10^{-7} to 1×10^{-11}. The salts may be in the form of lithium, sodium, potassium, copper, zinc, magnesium, calcium, barium, strontium, aluminum, lead, nickel, cobalt, manganese, chromium or iron salts, but I prefer to employ the alkaline earth metal salts, e. g., Ca, Mg, Sr and Ba. Of this group of weak acid salts, I prefer to use the oil-soluble metal salts of the phenols.

My invention is particularly novel in that the combination of salts of weak acids with the salts of the sulfonic acids has beneficial effect on the detergent properties of the oil and on any tendency toward development of corrosive conditions in the oil during use, which effect is not obtained when the substances are used by themselves. This effect can result from the use of smaller combined amounts of the materials than can be even approximated by the collective results from the separate use of larger amounts of the individual materials. Neither alone is sufficiently detergent. This will become evident from the examples given hereinafter. This is possibly explained by a theory that the soap of the weak non-carboxylic acid improves the colloidal property of the sulfonate in the oil and thereby enhances the detergency of the sulfonate. Also, the sulfonate may cause improvement in the functioning of the weak acid soap. Although the combination may be used in any good lubricating oil (including naphthenic base oils—low V. I. oils), it was found to be unexpectedly satisfactory in oils of a high viscosity index (V. I.), for example, oils with a V. I. of 85 to 95, possibly because of the unexpectedly high "solubility" or dispersion in this type of oil.

The term "viscosity index" is described in Chemical and Metallurgical Engineering, vol. 36, No. 10, pages 618–619 (1929). The term "soap," which is sometimes herein used to indicate the salts of this invention, indicates salts of high molecular weight materials possessing at least ten carbon atoms per molecule so as to impart good dispersibility or "solubility" in the mineral lubricating oil when in the form of salts of those metals whose salts are oil-dispersible or "soluble" as here required.

The sulfonated acids employed to prepare the sulfonate additive of this invention may be those synthetically produced as by du Pont, and obtainable on the market, or those obtained from the treatment of petroleum fractions, and also obtainable on the market as from Sonneborn Company of New York city. Acids of the latter type, which are valuable here, are the oil-soluble so-called "mahogany" acids.

The "mahogany" acids are well known to the industry. They are those sulfonic acids which are formed when lubricating oil fractions or similar petroleum oil fractions are treated with concentrated or fuming sulfuric acid. These so-called "mahogany" acids dissolve in the oil phase, whereas the so-called "green" acids are the water-soluble sulfonic acids which pass into the sludge. The mahogany acids may be recovered by treatment of the oils with sodium hydroxide to produce sodium sulfonate which is removed from the oil solution by means of the addition of an alcohol such as ethyl alcohol or propyl alcohol with heating whereupon the sodium sulfonates pass into the alcohol solution which is separated from the oil, the alcohol being then distilled off to leave the sodium sulfonates.

Sodium salts of suitable oil-soluble sulfonic acids of this type from petroleum are obtainable from the Sonneborn Company under the trade name "Petronate" which contains about 60% sulfonates and about 35% to 40% mineral lubricating oil.

In order to obtain the calcium sulfonate from this mahogany acid soap, the sodium or potassium sulfonate is dispersed in hot water and a solution of calcium chloride is added with agitation while maintaining the mixture near the boiling point of water. The result is the formation of calcium sulfonate in a sodium chloride and calcium chloride water solution. The mixture is cooled to coagulate the water-insoluble calcium soap, the water solution is drawn off, and the calcium sulfonate mass is washed with water in the cold. In order to eliminate all of the water and the remaining sodium chloride, from two or three volumes of mineral lubricating oil, based on the calcium sulfonate, are added to the calcium sulfonate mass and the mixture boiled until all of the water is eliminated, the temperature being finally raised to about 325° F. or within the range of perhaps 275° F. to 375° F. During this treatment, the calcium sulfonate passes into solution in the oil, the water is driven off and the residual sodium chloride which crystallizes in the oil from the water droplets is then removed by filtering the hot oil solution. Incidentally, any other inorganic salts including excess calcium chloride will have crystallized and been removed along with the sodium chloride.

In one instance 900 grams of the sodium salts of oil-soluble petroleum sulfonic acids were dispersed in 3000 grams of water and brought to a boil. To this was added 225 grams of calcium chloride dissolved in 1000 grams of water. The batch was agitated vigorously. The excess water was then expressed from the mass thrown down, and the mass washed with cold water with removal of the excess wash water. To the washed soap mass 2000 grams of lubricating oil such as described herein was added and the batch dehydrated as above. After adding 75 grams of a finely ground diatomaceous earth and heating to 300° F., the batch was filtered.

The resultant material is an oil concentrate containing from 25% to 35% of the oil-soluble petroleum sulfonic acid soap of calcium which otherwise may be referred to here as a calcium mahogany acid soap, or calcium mahogany soap. This concentrate is liquid at normal temperatures, is free from water and mineral salts and, while it has an increased viscosity over the original oil, it has no typical grease-like characteristics, that is, it is free from all gel structure typical of greases. The soap is contained in the oil in what appears to be a state of perfect dispersion approximating possibly a true solution, which appears to be neutral and without the presence of any appreciable acid number.

For the present combination, sufficient of this concentrate will be employed to impart to the oil a sulfonate content of around 0.3% to 3%, approximately 1% representing an average use. These salts are readily dispersible in paraffinic base oils of high viscosity index as well as in naphthenic base oils, in proportions larger than required in the final composition. Even the concentrate may be prepared with a high viscosity index oil.

Whereas the sulfonate additive is used primarily for its detergent properties, the weak noncarboxylic acid soap is added to activate or increase the detergent properties of the sulfonate as well as to impart resistance to the development of corrosive conditions affecting the particularly corrosion-sensitive bearings. This second additive ordinarily may be used in percentages somewhat under those for the sulfonate. For example, if 0.8% to 1% of calcium sulfonate is used, then about 0.5%, or apparently sometimes as low as 0.2% or 0.25% of the weak-acid soap, will ordinarily be used. This is especially true of the phenol soaps.

By the term "phenols" it is meant to include not only the hydroxy aromatic ring compounds in which an hydroxyl group is directly attached to a carbocyclic aromatic ring, but also heterocyclic compounds in which the hydroxyl group is attached to a ring containing a sulfur, oxygen, nitrogen or other atom, said compound being capable of forming an oil-soluble metal salt. It is to be further understood that the term "phenols" includes not only monohydroxy but polyhydroxy compounds and those in which more than one ring is present, such as naphthols and the like. In addition to the hydroxyl group or groups the ring is preferably substituted by one or more other substituents, for example, an alkyl group or a sulfur atom linking two phenolic radicals together such as in di-para-tertiary-amyl diphenol sulfide, the only limits being that the compound must have a sufficient low ionization constant as defined, must be non-carboxylic and must contain at least one hydroxyl group of sufficient acidity to exhibit an ionization constant within the limits described above, and must form an oil-soluble metal salt. For the sake of simplicity in the following specification and claims, these compounds will be referred to merely as the metal salts or metal soaps of phenols.

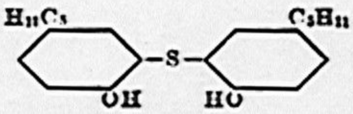
Although it is not definitely known why the metal salts of phenols act to prevent corrosion of alloy bearings, and I do not wish to be bound by the theory, it is presumed that this inhibiting power is at least in part connected with the ability of the salts to react with the corrosive acids formed in the lubricating oil during use to produce non-corrosive metal salts of the corrosive acids, thereby liberating the non-corrosive, weakly acidic phenolic compounds. As an indication of the manner in which the above mentioned compounds are presumed to react to neutralize corrosive acidity, thereby rendering the oil noncorrosive, the following exemplary equation may be given:

$$R-O-M+HX \qquad R-O-H+MX$$

In this equation R represents a hydrocarbon radical, M represents a metal, X represents the acid radical of a corrosive acid, and H represents a replaceable or acidic hydrogen atom.

The oil-soluble metal salts, such as the calcium salts of the phenolic compounds are employed in the lubricating oil in small amounts from about 0.2% up to about 3%, but in amounts less than that required to cause any undesirable thickening of the oil, the optimum apparently being from about 0.5% to 1.5%.

A specific usable phenolic material of which I have produced the calcium salt is di-para-tertiary-amyl diphenol sulfide, in which the amyl group is now believed to be para to the hydroxy position of the phenol, and which apparently has the formula:



Alkyl hydroxyl phenyl thio ethers of this type are described in the Mikeska at al. Patent, No. 2,139,766 and other similar compounds and their method of preparation are given in Mikeska et al. Patent, No. 2,139,321.

A phenolic material believed to be kindred to that of the Mikeska et al. Patent, No. 2,139,766, is at present obtainable on the market under the trade name "Paranox" which is available from the Standard Oil Development Company of Elizabeth, N. J. This material, apparently, is in general composed of mixed poly-alkyl polyphenol sulfides, that is, poly-alkyl substituted hydroxy phenyl thio ethers containing, for example, a thio ether of butyl phenol which may be designated as Bis (2-hydroxy-4-butyl phenyl) sulfide. This commercial product apparently contains various materials of the following general formulas:

$$R(C_6H_3OH)S(C_6H_3OH)R$$

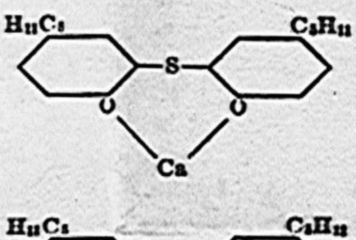
and

$$R(C_6H_3OH)S(C_6H_2OH)R.S(C_6H_3OH)R$$

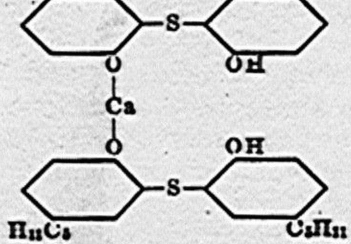
In these formulas and similar formulas herein, S, in general, indicates one sulfur atom, but in some molecules two or more sulfur atoms may be represented, the major proportion, however, preferably being monosulfides; R is an alkyl group preferably containing four or five carbon atoms (butyl or amyl) but may contain more, which groups may be different for the different components of the mixture or may be different for the two or more benzene nuclei in one of the components. R, S and OH may occupy any of the possible positions in the benzene rings. The material may contain higher polymers, such as indicated in the second formula above, and even more extensively polymerized materials.

In practicing this phase of the invention commercially, the soaps of suitable phenolic compounds, such as the calcium soaps of diamyl, or di-butyl, or other alkyl, or mixture of alkyl, phenol sulfides above indicated, may be formed without particular difficulty by any suitable procedure, as will be obvious to the skilled chemist. For example, the calcium salts of these compounds may be conveniently obtained by first adding the starting material to any approximately equal quantity or even greater quantity of a suitable lubricating oil, such as a naphthenic base mineral oil, or preferably for the purpose of the invention a paraffinic base oil of high V. I., having good solvent properties for the phenolic material and for the salts to be produced. This mixture is then commingled with hydrated calcium oxide and a small proportion of water, followed by heating to about 300° F. with agitation for a time sufficient to insure neutralization and dehydration. The resultant mixture is filtered to remove solids, such as excess calcium oxide. The ash (calcium content) has been increased when desired by first heating to only about 200° F. to 210° F. for a time to insure complete admixture and partial neutralization, the mixture being then cooled to 150° F. to 170° F. and a small quantity in the order of about 3% of 95% alcohol added, and the temperature of the mix then raised to the previously mentioned temperature of 300° F. Another method employed has been to neutralize the alkyl phenol sulfides with sodium hydroxide and then by metathesis, with calcium chloride or the like, convert the sodium salt to the calcium salt. In preparing for a treatment of the "Paranox" described above as probably comprising a mixture of alkyl phenol sulfides, about 20% of the sulfides should appear in about 80% of lubricating oil having good solvent power for the soap. Otherwise, a greater proportion of lubricating oil is added as its soap-dissolving power decreases. The above described neutralization to produce the calcium salts thereof is more or less easily accomplished due to the fact that the pure phenolic material has been found to have an acid number of about 98. The other alkaline earth metal salts may also be readily produced in a similar manner. In preparing soaps of other metals previously mentioned, the metathesis procedure just mentioned may be preferable as will be readily determined by the skilled chemist.

These salts or soaps are readily soluble in lubricating oils in the required proportions and yield alkaline solutions. Representative salts thus produced appear to have the following structural formulas:



and



Or the alkyl may be a butyl group, or a group of more than five carbons, thereby to impart good oil-"solubility" or dispersibility. Also the alkyl positions may be para to the sulfur or in any other position on the rings.

These materials thus may be viewed as sulfides, or as thioethers, or as metal oxides or as phenolates.

In preparing lubricating oil according to the present invention, the soaps or the soap-oil mixtures are stirred into an appropriate mineral lubricating oil and solution or dispersion is effected by agitation, a slight elevation of temperature being produced if desired to facilitate the operation.

Then the calcium soaps of these two types of compounds are dissolved in the lubricating oil to yield in the final product a total soap content of from about 0.5% to 3%, the detergent action of the resultant lubricating oil is sufficient to overcome or prevent the deposition of objectionable amounts of said resinous and varnish-like materials, in Diesel engines and the like. At the same time these quantities of soaps are insufficient to increase substantially the original viscosity of the mineral base lubricating oil. The ratio of the quantity of the phenol soap to the quantity of the sulfonic acid soap may be varied, but preferably approximately equal quantities of the two compounds are generally used, that is quantities to yield substantially equal amounts of ash figured as $CaSO_4$. The two soaps employed in conjunction with one another give excellent detergency characteristics to the oil, while the phenol soap itself acts also as an alkaline reserve to prevent the formation of, or neutralize the effects of, the objectionable corrosive acids which attack detrimentally the highly corrosion-sensitive alloy bearing of the copper-lead type.

In addition to the general detergent functions of the soap which are imparted to the oil by the calcium phenol soap, the presence of the sulfur in the soap molecule tends to impart to a non-sulfur-containing lubricating oil, extreme pressure characteristics, and the small percentage of actual sulfur present in the soap further acts to impart in itself some anti-corrosive properties.

In addition to the calcium soaps of the phenol thio-ethers indicated, soaps of other metals of the alkylated phenol thio-ethers may be employed, such as those of barium, magnesium and also zinc and aluminum, the important requirements being good solubility in the chosen mineral lubricating oil, detergent properties and freedom from appreciable viscosity increase when used in detergent quantities such as the indicated range from about 0.25% to 2.0% or possibly 3%. Also, these metals appear to possess less undesirable catalytic activity than some of the heavier metals such as lead. Soaps of other phenol ethers, such as alkyl-substituted phenol selenium or tellurium ethers or the like containing other suitable sulfur substitutes in the ether position, are also within the scope of this invention for some uses where possessing sufficient solubility or dispersibility in oil and adequate detergent properties.

I have also found that an alkyl phenol such as amyl phenol can be condensed with formaldehyde under appropriate conditions to form a viscous polymer in which free hydroxyl groups of the phenolic type are present, and that appropriate oil-soluble soaps such as calcium soaps can be produced therefrom. For example, I have manufactured the condensation product of p-tertiary amyl phenol with formaldehyde employing both an acid catalyst and a basic catalyst. Where employing an acid catalyst I have used about 82 grams (0.5 mol.) of amyl phenol with from about 27 grams to about 49 grams (0.33 to 0.5 mol.) of 37% formalin (formaldehyde) with 2 ml. of 37% of hydrochloric acid. These materials were combined and refluxed for one hour where the higher molecular proportions of formaldehyde were used and up to three hours where the lower proportions of formladehyde were used. The refluxing temperatures were conveniently carried between about 200 F. and 212° F., any appropriate range obvious to the skilled chemist being suitable. When the condensation product was formed by refluxing as above, a thick liquid, resinous material was obtained when the smaller proportions of the formaldehyde were used and a more pasty material when the higher proportions of formaldehyde were used. In these cases the resultant liquid or paste as one part was then added to nine parts variously of SAE 20 and SAE 30 grades of California or naphthenic base mineral lubricating oils and high viscosity index (80 to 90 V. I.) paraffinic lubricating oils. (In some cases, there was added to this mass the water layer from the refluxing operation, this water being that of the formaldehyde solution.) To this mass there was then added 37 grams (0.5 mol.) of hydrated lime and the batch was agitated for two hours at temperatures ranging between 180° F. and 200° F., and then cooled. In some instances 60 ml. of ethyl alcohol was then added and the batch further agitated for about two hours at 180° F. Where alcohol is used there is less hydrolysis and the calcium content is increased. In each instance the temperature and time of treatment and the nature of the treatment was such as to effect conversion of the phenolic resin into the calcium soap thereof. In order to remove completely any unconverted lime and other solvents there was mixed into the batch a quantity of suitable filter aid such as ground diatomaceous earth (e. g. "Super-Cel") approximating the amount of the hydrated lime employed (about 37 grams), and the batch heated to 300° F. and filtered at about that temperature.

Suitable resins were produced in the same manner by employing 2 ml. of ammonium hydroxide containing 28% NH_3 as the catalyst instead of hydrochloric acid, the refluxing in these particular cases being slightly longer, the results apparently being equally satisfactory.

These products constituted 10% concentrates, that is 10% of the calcium soap of the phenolic condensation product with 90% of the naphthenic mineral lubricating oil mentioned. These concentrates were dilutable in all degrees with either naphthenic base oils or paraffinic-type lubricating oils without precipitation of the soap after long standing. A higher soap concentrate can be obtained by using smaller amounts of oil, although the larger amounts render the mass more workable.

The same soaps have also been prepared directly by adding the hydrated lime to the original mass, thereby employing the lime as a catalyst. Thus 82 grams of amyl phenol, 328 grams of the mentioned 600 viscosity mineral oil and 37 grams of hydrated lime were charged into a closed flask, having a sealed agitator and a reflux condenser, and heated to 150° F. at which temperature the amyl phenol was melted. To this heated batch about 30 grams of 37% formaldehyde was introduced through the reflux condenser. Here the batch was refluxed at between 200° F. and 212° F. for about six hours. The product was then transferred to open vessel and agitated with 60 ml. of ethyl alcohol at 180° F. for 2 hours and filtered at 300° F. with the filter aid known on the market as "Super-Cel" which is ground diatomaceous earth.

By this last described procedure formation of the phenolic condensation product with the formaldehyde and its conversion into the calcium soap thereof was effected directly in one operation to yield a concentrate containing 20% soap.

Other alkyl phenols than amyl phenols where the alkyl group contains at least 4 carbon atoms such as butyl, hexyl, octyl, monyl, decyl and others of 11 and more carbon atoms per group may be employed as has been indicated above, and other metals than calcium, particularly the other alkaline earth metals and the light metals aluminum and zinc, may be employed in the formation of the oil-soluble metal soaps. These soaps may be considered either as phenolates or as metal oxides in which the rings carry alkyl groups to insure sufficiently high molecular weight to render them oil-soluble.

Further, I have found that stearyl alcohol can be condensed with phenol (monohydroxy benzene) in the presence of concentrated sulfuric acid to form a compound believed to be parastearylphenol. Soaps are prepared in manner similar to that described above. The metal alcoholates, such as aluminum, zinc, calcium and sodium alcoholate, where the alcohol consists of a hydroxy group attached to an alkyl, aralkyl, or cycloalkyl radical, also have value in use with the sulfonic acid soaps. The stabilizing effects which these alcoholates alone impart to lubricating oils are described in the Shoemaker et al. patents, Nos. 2,057,212, and 2,125,961, but the combination of these alcoholates with the metal sulfonates not only increases the inherent detergent properties of the metal sulfonates, but also the alcoholates retain their beneficial stabilization characteristics. These materials also may be considered as metal oxides.

In the foregoing disclosure, the oil-soluble metal soaps of phenolic compounds have been preferred for use along with the sulfonates, but it is to be understood that the oil-soluble metal soaps of other weakly acidic, non-carboxylic compounds having ionization constants as specified above, are equally satisfactory and are within the scope of the present invention. As group examples of other such weakly acidic, non-carboxylic materials, the phenols, thiophenols, other enols, oximes, sulfonamides and the like were previously mentioned.

The following are specific examples in addition to those given above of suitable acidic materials from the classes just mentioned, and include aryl and alkyl substituted phenols and others of the classes mentioned.

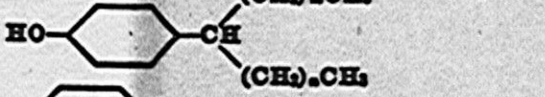
In these instances the designation of the various groups by *m* and *n* is to provide the respective molecules with sufficiently high molecular weights to yield soaps of good solubility in oil. Where the molecule has two rings, it appears that the molecular weight of a soap of good oil-solubility is around 350 or higher. There may be some instances where a soap of as low as 300 molecular weight will be sufficiently oil-soluble, but such apparently is not true of all cases. In general the same lower limits of molecular weights has been taken as representative of soaps of suitable acidic materials of other chemical configuration. Varying values for *m* and *n* also indicate complexity in the molecules, which appears to be a desirable feature imparting greater oil-solubility. Branching appears to promote oil-solubility. Further, mixtures of different molecules in which the alkyl groups vary appear to have better oil-solubility than materials representing a single molecule. Thus, these materials may contain alkyl groups (or aryl groups where indicated) in which *n*, or *m* and *n* combined, may be smaller when the molecules otherwise have relatively high molecular weights, and larger when the molecules otherwise have relatively lower molecular weights. In general it may be said that *m* and *n* will have values from zero to perhaps as high as 20 or possibly higher for molecules otherwise of low molecular weight, and from zero to perhaps about 10 for molecules of otherwise higher molecular weight. Where different groups appear in the same molecule as indicated by the presence of both *m* and *n*, the number of one will decrease as the other increases at least toward the upper limit, whereby to avoid undesirably high molecular weights.

The following examples of these materials are arranged according to groups above mentioned:

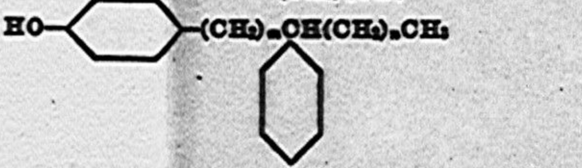
1. *Phenols*

(*a*) Monohydroxy phenols

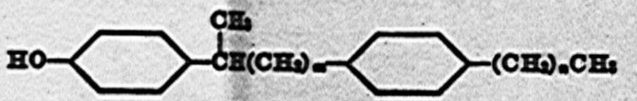
1.



2.

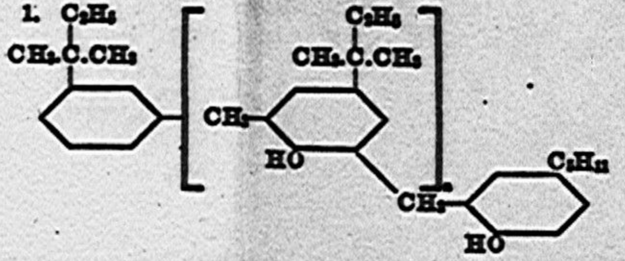


3.



(*b*) Polyhydroxy phenols

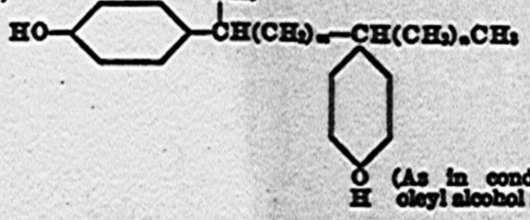
1.



(condensation product of p-tert. amylphenol and formaldehyde.)

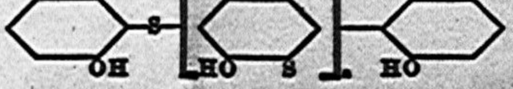
I(*b*)

2.



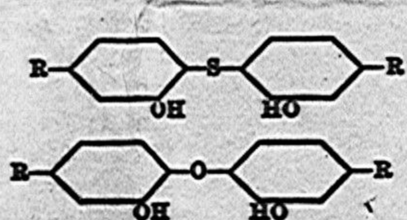
(As in condensation of oleyl alcohol and phenol)

3.

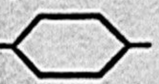


(R may represent groups from propyl to decyl for example)

4.

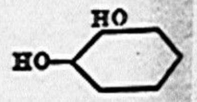


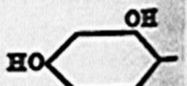
Here, R represents either an aryl group, such as



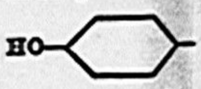
or an alkyl group such as amyl, butyl or other group between propyl and decyl, or an ester group or the like. Also, at least one R may represent a halogen such as chlorine or other suitable film strength or kindred substituent.

5. Products of the type in all previous examples in which



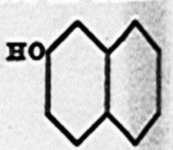


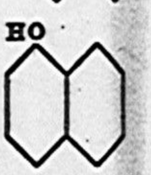
and the like appear in place of

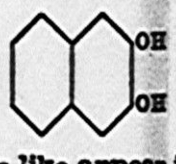


(c) Condensed ring phenols

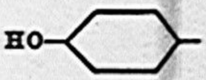
1. Products of the type in (a) and (b) above in which





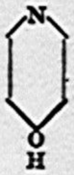


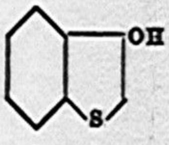
and the like appear in place of

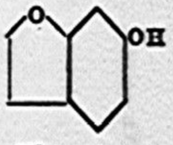


(d) Heterocyclic phenols

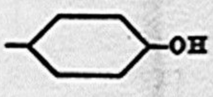
1. Compounds of the types in (a), (b), (c) above, in which







and the like appear in place of



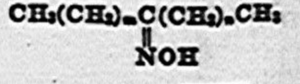
II. *Thiophenols*

An OH group in the phenol compounds of Group I is replaced with SH.

III. *Oximes*

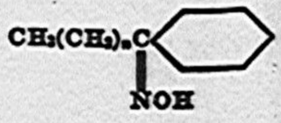
(a) $CH_3(CH_2)_nCH=NOH$

(b)

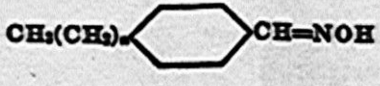


$CH_3(CH_2)_n\underset{\underset{NOH}{\|}}{C}(CH_2)_nCH_3$

(c)

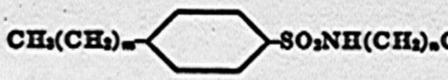


(d)

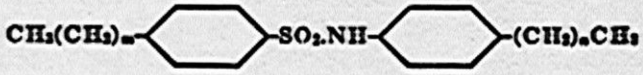


IV. *Sulfonamides*

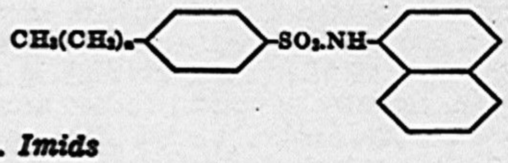
(a)



(b)

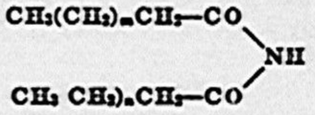


(c)

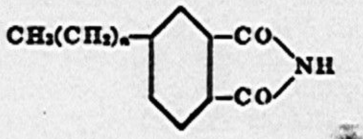


V. *Imids*

(a)

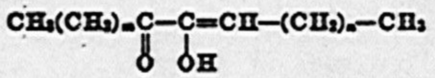


(b)



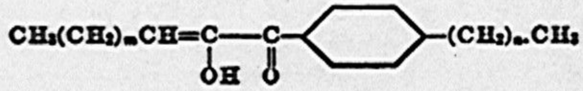
VI. *Enols*

(a)



$CH_3(CH_2)_n\underset{\underset{O}{\|}}{C}-\underset{\underset{OH}{|}}{C}=CH-(CH_2)_n-CH_3$

(b)



Oil-soluble soaps or salts of these various materials may be prepared in manners as outlined above and added in indicated proportions to yield the type of product described and claimed. Important requirements are good solubility of the soaps in oil in the proportions required, without substantial increase in the viscosity of the base oil, and low ionization constants as above defined with consequent freedom from development of corrosive conditions in the oil during engine use which is presumed to be due to the indicated neutralizing character of the soaps which may be termed "reserve alkalinity."

The salts or soaps of these materials from the indicated metals, may be considered as metal oxides as well as the phenolates, or ethers, or thio ethers as the case may be. They will be used with the indicated sulfonates in the percentage ranges previously indicated, but not sufficient to promote appreciable viscosity increase where to be used in internal combustion engines. As specific examples of the preparation of the combination of the invention and the effect of some of the hereindescribed materials, the following are presented:

One thousand grams of the above-mentioned oil-soluble sodium sulfonate in oil, known as "Petronate," produced by the Sonneborn Company, was agitated in 4000 cc. water during the addition of 220 g. $CaCl_2$ in 1000 grams water. After complete metathesis, the product was washed and added to 4000 grams of a propane dewaxed highly solvent-treated highly-paraffinic SAE 30 high viscosity index mineral lubricating oil (about 90 V. I.) together with about 25 grams of lime to insure complete neutralization. The mixture was dehydrated at 300° F. and filtered at 300° F. to give a filtrate component (I) which was a dispersion of 600 grams of oil-soluble calcium sulfonate in 4400 grams of oil.

To prepare the second additive for my composition, 2000 grams "Paranox" (amylphenol thioether) obtained from the Standard Oil Development Company, was mixed with 500 grams lime, 440 ml. 90% isopropanol and 8000 grams of the same high viscosity index (90 V. I.) SAE 30 motor oil mentioned in the above example. The mixture was agitated for two hours at 180° F., dehydrated for thirty minutes at 330° F. with 500 grams of diatomaceous earth to give component (II) consisting of 2000 grams of calcium soap of alkyl phenol thioether together with some unreacted alkyl phenol thioether in 8000 grams of oil.

These two preparations were added to a similar high V. I. solvent treated SAE 30 lubricating oil in quantities to give a composition containing 0.8% by weight of calcium sulfonate and 0.5% by weight of calcium soap of alkyl phenol thioether. This composition, on testing in Diesel engines running with a high load, proved to have excellent detergency properties. After the usual period of 100 hours, the piston was free of lacquer deposit, and all of the rings were free and in good condition. The corrosion of copper lead bearings was also at a minimum.

However, using the same high V. I. motor oil to which had been added 1.2% by weight in one case and 2.5% by weight in another case of the calcium sulfonate by means of component I, the products showed relatively poor detergency action in Diesel engines. The use alone of about 2.0% by weight of the described calcium soap of alkyl phenol thioether by means of component (II), whose active ingredient was probably calcium diamyl-diphenol sulfide, in a higher V. I. oil gave a correspondingly poor lubricant for severe service engines. At the end of forty hours deposits had appeared on the ring lands and the rings were beginning to show symptoms of sticking, and on the piston skirt a quantity of a varnish-like material was noticeable.

The above two-component composition is illustrative of the various compositions which can be made under the above disclosures employing sulfonates and any other weak-acid soap having the "reserve alkalinity" property.

It will be understood that examples are given for the purpose of illustration and not as necessarily limiting beyond the requirements of the prior art.

I claim:

1. Mineral lubricating oil containing a small proportion of oil-soluble petroleum sulfonate and a small proportion of the salt of a weak non-carboxylic organic acidic material having an ionization constant not exceeding about 5×10^{-6}.

2. A mineral lubricating oil containing a small proportion of oil-soluble soap of the oil-soluble sulfonic acids obtained by sulfonating petroleum fractions, together with a small proportion of oil-soluble salt of weak non-carboxylic organic acid having an ionization constant not exceeding about 1×10^{-6}.

3. A lubricant comprising mineral lubricating oil and a small quantity of each of (1) an oil-soluble metal soap of a weak non-carboxylic organic acid having an ionization constant less than 5×10^{-6}, and (2) an oil-soluble metal soap of oil-soluble sulfonic acids from petroleum.

4. A mineral lubricating oil containing a small quantity of an oil-soluble metal soap of a substituted phenol together with a small quantity of an oil-soluble metal petroleum sulfonate, the combination of these two substances in the lubricant having superior detergent and anti-corrosive properties which the substances do not possess alone.

5. A lubricant according to claim 3 wherein each of the soaps is present in amount between about 0.25% and about 3%.

6. A lubricant according to claim 4 wherein each of the soaps is present in amounts between 0.3% and 3%.

7. A lubricant according to claim 3 wherein the soaps are alkaline earth metal salts.

8. A lubricating oil according to claim 4 wherein the soaps are alkaline earth metal salts.

9. A lubricating composition as in claim 3 wherein the weak non-carboxylic organic acid soap is the soap of an alkylated phenol thioether oil is one having a viscosity index above about 80.

10. A lubricating composition according to claim 4 where the mineral oil has a high viscosity index.

11. A lubricating oil containing a small quantity of oil-soluble metal alcoholate together with small quantity of oil-soluble metal salt of sulfonic acid from petroleum.

12. A lubricant comprising mineral lubricating oil, a small proportion of an oil-dispersible aryl metal oxide and a small proportion of an oil-dispersible metal petroleum sulfonate.

13. A lubricant according to claim 12 wherein the lubricating oil is high viscosity index highly paraffinic mineral oil.

14. A lubricant according to claim 12 wherein the metal of the additives is an alkaline earth metal.

15. A lubricant comprising mineral lubricating oil having a viscosity index of at least about 80 containing a small proportion of oil-dispersible metal petroleum sulfonate and a small proportion of oil-dispersible metal phenolate.

16. A lubricant according to claim 12 wherein the aryl oxide contains substituents increasing the carbon atoms to at least ten per molecule.

17. A lubricant according to claim 1 wherein the weak acid contains at least ten carbon atoms per molecule.

18. A normally liquid lubricant comprising a mineral lubricating oil having a viscosity index at least as high as about 80, a small proportion in the order of 1% of an oil-soluble calcium soap of sulfonic acids derived from sulfonating petroleum fractions, and a small quantity in the order of 0.5% of an oil-soluble calcium salt of an alkylated hydroxy phenyl thio ether.

19. A lubricant according to claim 3 wherein the soaps are salts of metals other than alkaline earth metals.

20. A lubricating oil according to claim 1 wherein the salts are those of alkaline earth metals.

21. A lubricating oil according to claim 1 wherein the salts are salts other than alkaline earth metals.

22. A lubricating oil according to claim 1 wherein the base oil is an oil having a viscosity index of at least about eighty.

23. A lubricant according to claim 3 wherein the base oil is one having a viscosity index of at least about eighty.

24. A lubricant according to claim 3 wherein the salts are salts of heavy metals.

CHESTER E. WILSON.

8

CERTIFICATE OF CORRECTION.

Patent No. 2,280,419. April 21, 1942.

CHESTER E. WILSON.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 7, second column, lines 14 and 15, claim 9, strike out "oil is one having a viscosity index above about 80"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 4th day of August, A. D. 1942.

Henry Van Arsdale,
(Seal) Acting Commissioner of Patents.

APPELLANTS' BRIEF.

In the

United States Court of Appeals For the District of Columbia

United States Court of Appe
For the
District of Columbia Circuit
FILED MAR 21 1950
Joseph W. Stewart
CLERK

Appeal No. 10,520.

PETER A. ASSEFF AND THE LUBRI-ZOL CORPORATION,
Appellants,

v.

JOHN A. MARZALL,
Commissioner of Patents,
Appellee.

APPEAL FROM THE JUDGMENT OF THE DISTRICT COURT OF THE UNITED STATES FOR THE DISTRICT OF COLUMBIA.

OSCAR C. LIMBACH,
Leader Bldg., Cleveland, Ohio,
Attorney for Plaintiffs-Appellants.

ALMON S. NELSON,
604 Warner Bldg.,
Washington, D. C.,
Of Counsel.

THE GATES LEGAL PUBLISHING CO., CLEVELAND, OHIO—MAIN 1-5647

INDEX.

I. Jurisdictional Statement 1

II. Statement of the Case 2

1. The Invention 2
2. The Grounds of Rejection 3

III. Statement of Points 5

IV. Summary of Argument 6

V. Argument 7

1. The Prior Art 7
   - a. Salzberg 2,063,629 8
   - b. Wilson 2,280,419 9
   - c. McNab 2,289,795 10
   - d. Cook 2,344,392 12
   - e. Asseff 2,261,047 15
   - f. The Teachings of the Prior Art Cannot Be Combined to Spell Out an Anticipation of the Claims in Issue 15
   - g. Miscellaneous Grounds of Rejection 17
2. Asseff's Reduction to Practice 20
3. The 1st Point of Error: "The Preparation and Use of the Capryl Ester Was a Logical and Obvious Adaption of the Teaching of the Prior Art" 22
4. The 2nd Point of Error: "Salzberg Indicates the Equivalency of the Calcium and Zinc Salts" 23
5. The 3rd Point of Error: "Their Production (Zinc Dicapryl Di-Thiophosphate) and Use Was No More Than Would Be Reasonably Expected From One Skilled in the Art and Did Not Constitute Invention" 23

6. The 4th Point of Error: "Asseff Was Following * * * Prior Art When He Added a Separate Detergent to (Zinc) Dicapryl Di-Thiophosphate Salt" 25

7. The 5th Point of Error: "Legally * * * the Effective Date of the Cook Patent (2,344,392) is July 11, 1941, Since the Two Applications (Serial No. 401,960 Filed July 11, 1941, and Serial No. 418,326 Filed November 8, 1941, and Matured Into Patent 2,344,392) Cover the Same Subject Matter" 26

8. The 6th Point of Error: "Wilson Patent Teaches Those Skilled in the Art to Utilize a Plurality of Additive Agents and the Parent Cook, et al. Application Disclosed That the Specific Heavy Metal Salts of the Dithiophosphoric Esters Disclosed Therein Should be Used With Other Materials. There is, Therefore, Nothing Inventive in These Claims" .. 27

9. The 7th Point of Error: "The Inclusion of This Material (Phenates), Old in the Art, With the Basic Compound of the Cook Patent, is Not New or Inventive" 27

10. The 8th Point of Error: "Plaintiff's Claims Fail to Ante-Date the Anticipatory Effect of the Cook et al. Patent" 28

11. The 9th Point of Error: "Plaintiff's Claim 9 Through 24 Lack Invention and Patentability Over McNab, Salzberg and Wilson" 32

VI. Summary 32

VII. Relief 33

Appendix B. Chronological Chart ....Attached

TABLE OF AUTHORITIES.

Cases.

Becket v. Coe, 69 App. D. C. 53, 98 F. (2d) 332 (1937)	6, 22
Dewey & Almy Chemical Co. v. Mimex Co., 124 F. (2d) 986 (CCA 2nd 1942)	22
Eibel Process Co. v. Minnesota Ontario & Paper Co., 261 U. S. 45, 67 L. ed. 523, 43 S. Ct. 322 (Sp. Ct. 1923)	22
EK, In re, 57 App. D. C. 203, 19 F. (2d) 677 (1927)	23
Hartford-Empire Co. v. Hazel-Atlas Glass Co., 59 F. (2d) 399	24
Skelly Oil Co. v. Universal Oil Products Co., 31 F. (2d) 427 (CCA 3rd 1929)	23
United Chromium Inc. v. International Silver Co., 60 F. (2d) 913 (CCA 2nd 1932)	24, 25

Statutes.

R. S. 4915, 35 U. S. C. Sec. 63	1

In the

United States Court of Appeals For the District of Columbia

Appeal No. 10,520.

PETER A. ASSEFF AND THE LUBRI-ZOL CORPORATION,
Appellants,

v.

JOHN A. MARZALL,
Commissioner of Patents,
Appellee.

APPELLANTS' BRIEF.

I.

JURISDICTIONAL STATEMENT.

In this action, brought under the provisions of Section 4915 R. S., U. S. C. Title 35, Sec. 63, Plaintiff, Peter A. Asseff, as the original applicant, and The Lubri-Zol Corporation, as assignee of Asseff's entire right title and interest in the said application, by a complaint (2A) filed in the U. S. District Court for the District of Columbia, sought to have the Court order the Defendant, the Commissioner of Patents, to issue a patent upon certain claims of the application of Asseff, Serial No. 457,746 filed in the U. S. Patent Office on September 9, 1942. From a judgment entered October 17, 1949 (22A) dismissing the complaint, Plaintiffs have filed this appeal (23A).

The original Defendant, Lawrence C. Kingsland, who resigned as Commissioner of Patents, was succeeded to that office by Defendant, John A. Marzall, and by order of the District Court (22A) the present Defendant was substituted for the original Defendant, Kingsland.

II.

STATEMENT OF THE CASE.

1. THE INVENTION.

The invention in issue is defined by claims 1, 5, and 9 to 24 inclusive, set forth in the complaint (3A-7A). It is a lubricating composition which is designed to withstand the type of severe service encountered, for example, in Diesel engines which operate at high temperatures and high loads. The base of the lubricating composition is a conventional lubricating oil, such as a highly refined mineral oil, to which has been added one or more chemical fortifying and improving agents. Claims 1 and 5 (3A-4A) are the only claims calling for a single addition agent, which is therein defined as the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide. When an alcohol is reacted with phosphorus pentasulphide (P_2S_5), the immediate reaction product is an acid ester which may be neutralized by replacing the acidic hydrogen atoms of the ester with a metal such as zinc. This is accomplished by reacting the acid ester with an oxide of the metal. The resultant product is commonly referred to as a di-thiophosphate, the particular material of the present claims being zinc dicapryl di-thiophosphate. Instead of being thus specifically identified by precise chemical nomenclature, this additive material is referred to in the claims as the product produced by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulphide. This reaction, which may be carried on by the simultaneous admixture of the three reagents, as specifically called for by claim 1, or by first preparing the acid ester and then reacting it with the metallic oxide, produces a reaction mass which is predominantly the di-thiophosphate above identified and various side reaction products. Since it is not necessary to separate the di-thiophosphate from the reaction mass, and since the entire mass can be used as such, the claims identify the additive as the reaction product.

The remaining claims can be divided into three groups. The first group includes claims 9 to 14 inclusive, and 17 to 24 inclusive. These are characterized by the required additional presence in the composition of a second additive which is generically defined in claims 9, 10, 12, 13 and 17 to 21 inclusive, as a "detergent"; in claims 11 and 14 as "a calcium salt of a petroleum sulphonic acid," and in claims 22 to 24 as a metallic phenate, which is more specifically identified in claims 23 and 24 as having been derived for an ester of salicylic acid such as lauryl salicylate.

The second group of multi-additive claims includes claim 15, in which the second additive is "a stable oil-soluble halogen bearing compound." The third group of multi-additive claims includes claim 16, in which the second additive is broadly defined as "a separate oil-soluble organic corrosion inhibitor."

Certain of the claims, such as 17, 19 and 21, define a composition comprising three components, namely the thiophosphate, the detergent, and a separate oil-soluble organic halogen compound.

2. THE GROUNDS OF REJECTION.

In the Defendant's answer (11A), four prior patents were specifically enumerated as anticipatory of the claimed invention. In the amended answer (12A), two additional patents were cited. The solicitor for Defendant apparently no longer relies on the Mussellman patent. It was not referred to by the Trial Court. It is believed, therefore, that the prior art now relied upon as anticipatory is the following:

Salzberg et al.,	2,063,629,	Dec. 8, 1936,
Wilson,	2,280,419,	April 21, 1942,
McNab,	2,289,795,	July 14, 1942,
Cook et al.,	2,344,392,	March 14, 1944,
Asseff,	2,261,047,	Oct. 28, 1941.

Copies of the foregoing patents are reproduced in the Joint Appendix 177A-201A.

At the trial the entire Patent Office record of the Asseff application was introduced as Plaintiff's Exhibit 43, the pertinent portions of which are reproduced in the Joint Appendix 114A-163A.

At the trial two witnesses testified, and their testimony is concerned principally with Asseff's development program which led up to the filing of the involved application, and by which testimony it was sought to be established that Asseff's completion of the invention, as defined by the claims, was prior to the effective date of any of the references which taught or suggested such invention. In order to facilitate the Court's consideration of the facts and to shorten the present brief, a chart has been prepared, attached to Plaintiff's brief, as Appendix B, and which shows chronologically the pertinent portions of Asseff's development program, and the work done by the patentees of the references relied upon as anticipatory of Asseff's invention.

III.

STATEMENT OF POINTS.

The following is an enumeration of points of error in the decision of the District Court upon which this appeal is based, references being made to page and line numbers of the Joint Appendix where such errors occur in the decision of the District Court.

1. "The preparation and use of the capryl ester was a logical and obvious adaption of the teaching of the prior art," (17A, lines 13 to 15).
2. "Salzberg indicates the equivalency of the calcium and zinc salts" (17A, lines 16 and 17).
3. "Their production (zinc dicapryl di-thiophosphate) and use was no more than would be reasonably expected from one skilled in the art and did not constitute invention" (17A, lines 22 to 24).
4. "Asseff was following * * * prior art when he added a separate detergent to (zinc) dicapryl dithiophosphate salt" (17A, lines 32 to 34).
5. "Legally * * * the effective date of the Cook patent (2,344,392) is July 11, 1941, since the two applications (Serial No. 401, 960 filed July 11, 1941, and Serial No. 418,326 filed November 8, 1941, and matured into Patent 2,344,392) cover the same subject matter" (19A, lines 26 to 28).
6. "Wilson patent teaches those skilled in the art to utilize a plurality of additive agents and the parent Cook, et al. application disclosed that the specific heavy metal salts of the dithiophosphoric esters disclosed therein should be used with other materials. There is, therefore, nothing inventive in these claims" (21A, lines 22 to 27).
7. "The inclusion of this material (phenates), old in the art, with the basic compound of the Cook patent, is not new or inventive" (21A, lines 34 to 36).

8. "Plaintiff's claims fail to ante-date the anticipatory effect of the Cook et al patent" (22A, lines 1 and 2).
9. "Plaintiff's claims 9 through 24 lack invention and patentability over McNab, Salzberg and Wilson" (22A, lines 3 to 5).

IV.

SUMMARY OF ARGUMENT.

The Cook patent is not an anticipation of the Plaintiff's invention because it is too late. The patent does not have an effective date as of July 11, 1941, which was the date of the parent Cook application, but is instead limited to the date of November 8, 1941 when Cook, et al. filed the application which matured into their patent. Actually, in their parent July 11, 1941 case, Cook, et al. did not disclose any specific zinc dithiophosphates, and with regard to the possibility of using any dithiophosphate in combination with a detergent, said that this could not be done since "each chemical tends to offset the action of the other."

The Cook application filed November 8, 1941 was filed by them after the publication of the Asseff patent which gave eleven examples of finished lubricants containing the combination of various detergents and zinc dithiophosphates.

None of the other cited art is an anticipation because it does not meet the test "would a man who was grappling with the problem solved by the patent attacked, and having no knowledge of that patent, if he had had the alleged anticipation in his hand, have said: 'That gives me what I wish.'"

The foregoing doctrine was cited by this Court with approval in *Becket v. Coe,* 69 App. D. C. 53, 98 F. (2d) 332 (1937). From an inspection of the chart in the appendix to this brief it will be observed that all of the remaining prior art relied upon by the Examiner is based on applications which were filed prior to the July 11, 1941 application of Cook, et al. Cook et al. are, therefore, a ready made example of one "who was grappling with the problem." At the same time Plaintiff, Asseff, was grappling with the problem, Cook, et al. were skilled workers in the art and their statements, as given in their July 11, 1941 application, are a much better basis to rely on in determining whether the steps taken by Asseff were invention than any reasoning which we might now indulge in, which is based upon knowledge after the fact.

Certain assumptions made by the trial court, such as for example, wherein the court held the calcium and zinc salts to be equivalent, are directly contrary to the facts of record in this case. Those items will be discussed in detail in the summary of our argument, wherein we will treat categorically the nine points enumerated above in Section III.

V.

ARGUMENT.

Before we take up for consideration the nine points of error on which this appeal rests, it is believed well to first consider in detail the actual teaching which is found in each of the prior art references, and to then show by a summary of Asseff's development program, wherein such program which led to the invention of the claims in issue, ante-dated any suggestion in the prior art of such invention.

1. THE PRIOR ART.

In considering the prior art at the time of the trial, the expert, Dr. Smith, was interrogated with regard to the technical aspects of each of the prior art disclosures, and his testimony with regard to such prior art will be referred to in the interpretation of such art.

a. Salzberg 2,063,629.

Dr. Smith's testimony with regard to this patent begins at about the middle of page 82A of the Joint Appendix and such testimony was to the following effect.

Salzberg, (190A), column 2, lines 1 and 2, states that the esters to which that invention relates, "contain at least one aliphatic chain of at least 10 carbon atoms." Capryl alcohol, as stated by Dr. Smith, contains 8 carbon atoms and therefore the organic portion of the characterizing addition agent to which the issue claims are directed, is quite different from the ester portion of the Salzberg compounds. Salzberg said that compounds containing less than 10 carbon atoms in the molecule would not work. Asseff, on the other hand, discovered that a compound containing the capryl alcohol group of 8 carbon atoms was highly effective. Moreover, as pointed out by Dr. Smith (83A), while Salzberg on page 192A, second column, beginning with line 17, states that the invention, "embraces the acid esters and their salts" and while a series of salt-forming elements or radicles are mentioned, *Salzberg does not give any example of the preparation of any salt nor does he identify any specific salt in his patent.*

As an indication of the fact that (a) the salts of the capryl alcohol product produce results different from those produced by the use of the longer chain alcohols as proposed by Salzberg; and that (b) the short chain or capryl alcohol product of the claims in issue is superior to the longer chain alcohol products of Salzberg, certain engine tests were run, the results of which are of record in the two sheets which comprise Exhibit 39. Dr. Smith's testimony with regard to that exhibit begins at the middle of page 77A and extends to the middle of page 81A. The first sheet of Exhibit 39 gives the results of a Chevrolet engine test on a composition in accordance with the Asseff invention. The second sheet of Exhibit 39 gives the results of an identical test on a compound which was identical with the Asseff compound, excepting that the alcohol used in the preparation thereof contained 18 carbon atoms (as in Example 2 of Salzberg) instead of the 8 carbon atoms of the capryl alcohol used by Asseff. The results of that test show conclusively that there is a real difference and it is, of course, for this reason that the claims in the present case are restricted to the capryl alcohol compound. That material was found to be so outstanding that the application involved in these proceedings is directed specifically to the material using capryl alcohol as one of the starting materials.

Salzberg is, therefore, not an anticipation of the claims in issue because he stated unequivocally that the alcohols from which the products were to be produced should have at least 10 carbon atoms, whereas Asseff's product contains only 8. Asseff's product is superior to the Salzberg products and from the standpoint of Salzberg's teaching, the results secured by Asseff are entirely unexpected. In addition to the above, Salzberg does not disclose in his patent the specific composition of *any* zinc salts.

b. Wilson, 2,280,419.

Dr. Smith's testimony with regard to this patent begins at about the middle of page 84A and extends over to the middle of page 85A. This Wilson patent, as explained by Dr. Smith, is directed to a two-component composition, the first component of which is defined as "an oil soluble petroleum sulphonate"; and the second as "the salt of a weak non-carboxylic organic acid material having an ionization constant not exceeding about 5×10^{-6}." It is this second component which the Patent Office and the Trial Court presumably intended to construe as the equivalent of the zinc salt of the claims in issue. The Wilson patent fails as an anticipation for two reasons. First, as explained by Dr. Smith in his answer beginning at the bottom of page 84A, whereas the Wilson patent is directed to salts of weak acids such as those derived from phenol, the acid produced by the reaction of capryl alcohol with phosphorus pentasulphide is a strong acid, far outside of the range indicated by the ionization constants of Wilson. In the second place, whereas the zinc salt of the reaction product with capryl alcohol and P_2S_5, or at least the major portion of such reaction product or salt, may be properly identified as a di-thiophosphate, *Wilson does not disclose anywhere in his patent any di-thiophosphates.*

c. McNab 2,289,795.

Dr. Smith's testimony with regard to this patent begins at the middle of page 85A and extends over to the middle of page 86A. The invention of this patent is most clearly stated in the specification thereof, page 185A, column 1, in lines 29-31, where it says:

> "It has now been found that certain mixed soap compositions are very much superior to compositions containing a single soap."

McNab found that if he had present in the same oil a plurality of soaps of different metals, improved results would be secured. On page 185A, first column in the paragraph beginning with line 29, he divides the usable metals into three groups:

> "* * * the first comprises calcium and magnesium soaps which appear to be interchangeable, although they may be used together if desired. The second type of soap is that of aluminum and the third of nickel."

McNab did not contemplate the use of any zinc salts. As pointed out by Dr. Smith, McNab, on page 187A, column 2, at line 70, mentions the compound calcium di-octyl di-thio-phosphate. At the top of column 1, page 188A, McNab states:

> "Many of the compounds listed are only sparingly oil soluble and can consequently only be employed with the assistance of a mutual solvent, etc."

This is confirmed by the data of record in this case, see Exhibit 24, Sample 601-305, 601-306, and 601-307, which were tests of 2% concentrations in three different oils of compound 601-304, i.e., the corresponding calcium salt of the acidic reaction product of capryl alcohol and P_2S_5. Those tests showed that the calcium salt was insoluble in each of the three oils in which it was tested, whereas the corresponding tests using the zinc salt showed the latter to be soluble. This is one of the decided differences between the compounds disclosed by McNab and the compound with which Asseff is concerned.

Another decided difference between the Asseff compound and the McNab compound is that the alcohol used by McNab was *normal* octyl alcohol, whereas the claims under consideration here are concerned with the *secondary* octyl or capryl alcohol. Dr. Smith, in his cross examination on page 88A, explained in detail the difference between the *normal* octyl alcohol used by McNab and the *secondary* or capryl alcohol used by Asseff. In his testimony on cross examination, in referring to the composition of Exhibit 52, which was a composition using the Asseff material, or that derived from capryl alcohol, and Exhibit 54, which was concerned with a composition identical with the previous one, excepting that the alcohol was normal octyl, Dr. Smith stated that the test results in the engine showed these materials to be quite different in their effect. The details of such difference is explained on page 93A.

In summary, the *normal* octyl compound, i.e. even in the form of the zinc salt instead of the calcium salt used by McNab gave a corrosion loss of 166 milligrams, as compared with a 69 milligram loss when using the composition of Asseff.

In conclusion with respect to McNab, therefore, that patent fails as an anticipation; first, because he does not disclose any zinc salts; and second, because he does not disclose the use of the capryl alcohol derivative to which the claims in issue are specifically directed.

d. Cook 2,344,392.

This patent does disclose the use in lubricants of the zinc salt of the reaction product of capryl alcohol and P_2S_5. Cook does not direct any of his claims specifically to zinc. In the specification of the Cook patent, page 183A, column 2, beginning with line 48, Cook mentions the fact that the di-thio-phosphates may be used in combination with detergents.

Our position with regard to the Cook patent is that Asseff made his invention as of a date prior to the invention date of Cook.

The filing date of the application which matured into patent No. 2,344,392 is Nov. 8, 1941. The earlier application Ser. No. 401,960, page 164A, previously referred to, was filed July 11, 1941. The July 11 application disclosed certain di-thio-phosphates. The only *di-capryl* di-thio-phosphate disclosed in the July 11, 1941 Cook application was *barium* di-capryl di-thio-phosphate shown as the first composition in Table II, page 172A. That July 11 application also contained the statement quoted at an earlier point in this brief to the effect that the di-thio-phosphates or inhibitors could not be used in combination with detergents because the effect of each chemical was to offset the action of the other. In considering the anticipatory effect of the Cook patent, it is important to trace the chronology of events in the summer and fall of 1941.

Prior to July 11, 1941, Asseff had run engine tests on the compositions of Exhibits 47-51 inclusive. These showed the production of desirable results from the use in a lubricant of the di-thio-phosphates which were made the subject matter of an Asseff application Ser. No. 404,361 filed July

28, 1941 and also the cooperative effect of the combined use of such di-thio-phosphates and various detergents.

On July 11, 1941 Cook et al. filed their application in which they stated that the heavy metal di-thio-phosphates were desirable for use, but that they could not be used with a detergent.

On July 28, 1941 Asseff filed his application Ser. No. 404,361 which has now matured into patent No. 2,261,047 giving a number of examples of the use in lubricants of di-thio-phosphates in which the alcohols used in the preparation of the same were cycloaliphatic alcohols. It is believed significant and of interest that at the time Asseff filed his first application on July 28, 1941, the McNab patent No. 2,289,795, previously referred to, had not yet issued, so that Asseff could not possibly have derived any suggestion from the McNab patent.

In the interval between July 28, 1941 and Nov. 8, 1941 Asseff ran further tests, including engine tests on the compositions of Exhibits 52, 53 and 54, the compositions of Exhibits 52 and 53 being within the scope of the claims here in issue.

On Oct. 28, 1941 Asseff's first application Ser. No. 404,361 issued into patent and the subject matter thereof accordingly became known to Cook and Thomas.

On November 8, 1941 Cook and Thomas filed their application Ser. No. 418,326 on which their patent No. 2,344,392 is based. In that application they now stated that the di-thio-phosphates *were* usable in combination with a detergent, although *no test data on such combined use is given,* indicating rather clearly that they had not had time to actually explore that combined use. It is also significant that *their patent does not claim the combination* as their invention.

In the interval between Nov. 8, 1941 and Sept. 9, 1942 Asseff continued his investigation which then led to the filing of his application Ser. No. 457,746, the application involved in the present proceedings.

Now with regard to the use in a lubricant of the zinc salt of the acidic reaction product of capryl alcohol and P_2S_5 (claims 1 and 5). This invention was *completed* by Asseff with his engine tests completed Oct. 3, 1941 on the composition of Exhibit 52. As previously pointed out, the Cook and Thomas application filed July 11, 1941, while mentioning certain di-thio-phosphates as desirable for use, did not mention specifically the use in a lubricant of the *zinc* salt of the reaction product of *capryl* alcohol and P_2S_5.

The July 11, 1941 Cook application did not come as close to the invention of the claims here in issue as did Asseff's earlier work in 1940 on the composition of Exhibits 47-51 inclusive and the work reported in his application filed July 28, 1941.

Stated in another way, Asseff on May 20, 1940, a whole year ahead of the earliest Cook and Thomas application, had run an engine test on the composition of Exhibit 49 in which the inhibitor was the zinc salt of octyl alcohol reacted with P_2S_5 and while in their July 11, 1941 application, Cook and Thomas had shown the preparation of barium-dicapryl di-thio-phosphate and zinc di-butyl di-thio-phosphate and zinc di-lauryl di-thio-phosphate, they had not prepared zinc di-capryl di-thio-phosphate, since that material was not included by them until disclosed in their application filed Nov. 8, 1941 and even then, no real data was given with regard thereto and no claims were drawn to that material. As to claims 1 and 5, therefore, which are directed to lubricating compositions containing specifically the zinc salt of the reaction product of capryl alcohol and P_2S_5, Asseff has shown a completion of the invention earlier than any date of record on behalf of Cook and Thomas. The party Asseff is unable to provoke an interference with Cook and Thomas as to the subject matter here in issue, because Cook and Thomas never claimed it as their invention.

As indicated above, the earliest disclosure of any combination of a thio-phosphate with any other additive in any of the Cook and Thomas applications was on Nov. 8, 1941, after the issue of the Asseff patent disclosing such combination, and by that time Asseff had completed not only the series of engine runs in 1940 on the subject matter of Exhibits 47-51 inclusive, but also had completed the engine run on the composition of Exhibit 52 which is the precise subject matter of the claims here in issue. Additionally, a test was completed by Nov. 2, 1941 by Asseff on the composition of Exhibit No. 53, which likewise is an example of the compositions covered by certain of the claims.

e. Asseff 2,261,047.

It is believed unnecessary to treat this prior patent in any further detail at this point since it is of interest only from the standpoint of the historical background of the present invention and with regard to that aspect of the case, all of the comments necessary have been made. It is a significant fact which should not be overlooked that the original Asseff patent issued Oct. 28, 1941 is not a statutory bar against the application here in issue, which was filed Sept. 9, 1942, i.e., less than a year after the issuance of the parent patent, so that to the extent that there is any suggestion of the present invention in the parent case, that was not dedicated to the public, since by the filing of the present application within a year from the date of issuance of the parent case, any such dedication is negatived.

f. The Teachings of the Prior Art Cannot Be Combined to Spell Out an Anticipation of the Claims in Issue.

As pointed out above in connection with the consideration of the Salzberg patent, he suggested the use in lubricants of thio-phosphate esters derived from alcohols which have at least 10 carbon atoms, whereas the alcohol used in the preparation of Asseff's product contains only 8. Moreover, Salzberg does not disclose the specific composition of any zinc salts.

As likewise indicated above, Wilson does not disclose any di-thio-phosphates and second, the acids used by Wilson in the preparation of his salts are relatively weak acids, a point which is particularly stressed by Wilson, whereas, as explained by Dr. Smith, the acidic material from which Asseff prepares his zinc salt is a strong acid.

McNab did not contemplate any zinc salts. He carefully listed the metals from which the mixed salts or soaps were to be prepared and zinc was not included.

From the foregoing it is difficult to see how the teachings of any of these patents can be combined. The disclosures of the three patents are so radically different that it is not seen how the teaching of any one of them may be combined with either or both of the others in order to spell out an anticipation of anything. The Trial Court did not indicate the specific basis on which he felt that the teachings of any of the prior art might be combined. It is improper to contend that Wilson and McNab disclose the use of corrosion inhibitors in combination with detergents and that in view of this teaching there would be no invention in adding these detergents to Salzberg's composition or in adding a specific detergent to a Cook composition.

In the first place, even if a detergent were added to the Salzberg composition, one would still not have the composition claimed by Asseff, since the thio-phosphate of Salzberg is one which is derived from an alcohol containing at least 10 carbon atoms, whereas Asseff's material is derived from an alcohol having 8 carbon atoms. It is believed ridiculous to suggest that it is *obvious* to add a detergent to the composition disclosed by Cook and Thomas in their July 11, 1941 application. Such application said specifically that the combination of a detergent with a thio-phosphate like the corrosion inhibitor of Asseff's was *inoperative* since "each chemical tended to offset the action of the other." That is a statement which was made by Cook and Thomas, skilled workers in the art, and is certainly entitled to more weight than any conjectural statement made now with knowledge after the fact derived from Asseff's own invention. It cannot be said that it is obvious to add a detergent to the Cook et al. composition when Cook et al. said that that combination would not work. This is believed to be a clear demonstration of the fact that that combination as called for by certain of the claims is a *real invention.*

g. Miscellaneous Grounds of Rejection.

At the time of the trial, counsel for the Defendant urged that certain of the claims were unpatentable because of the use therein of expressions such as "characterizing amounts." He urged further that certain of the claims did not specifically call for the inclusion in the composition of mineral oil.

As pointed out by us in our opening statement at the trial, page 25A, the business of the plaintiff company, The Lubri-zol Corporation, is to manufacture and sell the chemicals such as those identified by the claims in issue. These chemicals are then used by the major oil companies in admixture with mineral oil in the production of the finished lubricant composition. The thing which the plaintiff company sells is a concentrate. That concentrate may consist of the zinc salt in a rich solution in mineral oil or when combined with the other cooperating agents such as a detergent, the concentrate need not contain any oil since the oil adds to the bulk and weight of the concentrate and accordingly increases the shipping and handling costs.

It is true that if an inventor finds a new use for an old chemical he may not claim the invention in terms of the composition by a claim which calls merely for the old chemical plus a recital of the new use. When, however, the inventor produces a new admixture of old materials, composition claims are allowable unless that specific admixture or the equivalent thereof has been produced previously.

In each of the claims here in issue where the only additive material called for is the zinc salt, those claims each additionally require the presence of mineral oil. Each of those claims is directed to a combination of components which has never been produced before. That combination of materials is a lubricating composition in every sense of the word in that it is useful as a lubricant and is useful in the manufacture of other lubricants by the mere admixture therewith with mineral oil. There is no evidence of record that the concentrates called for by the claims do not possess lubricating properties. The chemicals used are very expensive as compared with oil. The principal reason for their use in such low concentrations is because richer concentrations, which would be more expensive, are not required to do the job. The specification of the Asseff application is quite specific as to the percentages which may be used to give satisfactory results and since the claims of a patent must be interpreted in the light of the specification supporting the same, it is quite clear what is meant by the term "a characterizing amount." While certain of the claims were objected to on this ground during the early stages of the prosecution of the application before the Examiner, he did not list this objection in his summary of objections in his Examiner's Statement to the Board of Appeals. Allowance of claims of the character referred to is necessary for full protection. Otherwise, infringement could be avoided by plaintiff's competitors by the sale of a rich concentration of the zinc salt in mineral oil which would then require the plaintiff to seek out and bring suit against each customer using such rich concentration in the manufacture of the more diluted embodiment of the invention.

The attorney for the Defendant at the time of trial objected to the inclusion in the claims of terms such as "corrosion inhibitor." The inclusion of a generic term in a claim is objectionable only if the meaning of that term is indefinite so that it is impossible to determine whether or not infringement exists. From a reading of the specification of the reference patents, the terms "detergent" and "corrosion inhibitor" are used frequently. The specifications of these patents were prepared by persons skilled in the art and to them the terms in question had a definite meaning. The materials thus identified are well recognized classes of chemicals and there is no known way in which they could be otherwise identified in the drafting of a patent claim. The invention which was made by Asseff was the discovery of the unexpected results produced by the use of the particular zinc salt. Not only did it give outstanding results when used alone, but unexpectedly also it gave superior results when used in combination with the other named materials. The invention is not concerned with specific percentages of the zinc salt, nor with the combination of the zinc salt with any specific detergent or corrosion inhibitor, although some are obviously preferred. Asseff was the first to ever produce a lubricating composition containing this zinc salt and he was also the first to discover that such zinc salt *in combination* with the other named cooperating addition agents gave *superior* lubricants. In the complete absence of art we see no reason why his reward should be limited by artificial and highly technical requirements which have nothing at all to do with the invention. The terminology used by the claims has a definite meaning. It is terminology commonly used by those skilled in the art and, therefore, those to whom the patent is directed will have no difficulty whatsoever in determining whether or not they are infringers.

2. ASSEFF'S REDUCTION TO PRACTICE.

Example 6 referred to in Exhibit 52, as well as the remaining examples identified on Exhibit 52, is a composition which is a combination covered not only by the broad claims in issue, but also by the claims which call for the combination of the zinc salt with a detergent. At this point it should also be noted that the second material in Example 6 is a phenate which is one of the detergents which was specifically claimed because it was one which gave outstanding results. We shall not burden the brief with a consideration of the exhibits which establish the preparation of the composition and its test. The data summarized in Exhibit 52 is believed to do this with sufficient particularity. It may be well to refer to Exhibit 40 for a moment because the data therein contained show not only the remarkable results secured from the use of the zinc salt derived from capryl alcohol, but also the superiority of that material over use of a similar material derived from normal octyl alcohol. At the end of 150 hours operation, the engine using the capryl alcohol product was in much better condition than the engine using the product derived from normal octyl alcohol. The piston was cleaner; the amount of sludge behind the piston rings was only 5% as compared with 90% filling and the milligrams corrosion of the bearing with only 69 as compared with 166. That outstanding performance, totally unexpected, was the discovery which is the invention here in issue. That was all done and completed while Cook and Thomas were still seeking the answer by the use of other alcohols and other metals.

Possibly one reason why Cook and Thomas were not able to more correctly evaluate the several materials with which they were working is they apparently never ran their compositions in an engine. They were seeking to evaluate these various compositions entirely on the basis of a laboratory test in which 300 cc. (less than a pint) of the lubricant was placed in a large test tube and air blown through the oil for a period of 70 hours while various metals were suspended in the sample. In their July, 1941 application, in Table II, page 172A, they give the results of tests on eight different compositions. In the first six, the metal was barium with a different alcohol for each example. In the last two, the metal was zinc with a different alcohol for each example. However, the last example uses di-lauryl alcohol which was the same alcohol used in the third example. A comparison of the results of those two examples is important. The barium di-lauryl di-thiophosphate increased the weight of the bearings by 7 milligrams, showing that the material was reactive with the bearing surfaces and built up a film thereon such as is required of a corrosion inhibitor. The corresponding zinc compound decreased the weight of the bearings by 4 milligrams, which would lead to the conclusion that instead of acting as a corrosion inhibitor, corrosion was probably taking place.

Now let us refer to the only table of data given in the Cook and Thomas patent, page 184A. Those data are limited to barium di-capryl compounds. This was undoubtedly due to the fact that the zinc compound in the earlier application showed up so poorly that it was discarded. While the zinc salt was apparently prepared just before the filing by Cook and Thomas of their Nov. 8, 1941 application, they never apparently bothered to run any tests on it, not even the oxidation and corrosion test-tube tests to which the barium compounds were subjected. This record of the Cook and Thomas applications shows conclusively that they had come to the conclusion that barium was the metal to use instead of zinc.

3. THE 1st POINT OF ERROR: "THE PREPARATION AND USE OF THE CAPRYL ESTER WAS A LOGICAL AND OBVIOUS ADAPTION OF THE TEACHING OF THE PRIOR ART."

The test to be applied in determining whether a prior art patent is anticipatory is that given in *Becket v. Coe,* 69 App. D. C. 53, 98 F. (2d) 332 (1937), which is to the effect that the prior art without more, must give the complete answer. The evidence of record referred to above shows that there is an unusual and unexpected superiority in the addition agent which comprises the combination of zinc as the metal and the capryl radicle as the ester forming substituent. It might be contended that this is a narrow step forward but it is many times the last narrow step which is entitled to as much recognition as the primary basic invention. In just such a circumstance the Supreme Court speaking through Mr. Chief Justice Taft in *Eibel Process Co. v. Minnesota Ontario & Paper Co.,* 261 U. S. 45, 67 L. ed. 523, 43 S. Ct. 322 (Sp. Ct. 1923)., said

> "His was not a pioneer patent, creating a new art; but a patent which is only an improvement on an old machine may be very meritorious and entitled to liberal treatment. Indeed when one notes the crude working of machines of famous pioneer inventions and discoveries, and compares them with the modern machines and processes exemplifying the principle of the pioneer discovery, one hesitates in the division of credit between the original inventor and the improvers; and certainly finds no reason to withhold from the really meritorious improver the application of the rule 'ut res magis valeat quam pereat,' which has been sustained in so many cases in this court."

Judge Learned Hand, in the case of *Dewey & Almy Chemical Co. v. Mimex Co.,* 124 F. (2d) 986 (CCA 2nd 1942) stated the same doctrine in somewhat different language as follows:

> "The patent law is aimed at animating a lower order of imagination and skill than that; more, it is true, than

> the ordinary rub of competition automatically brings out from competent workmen in the art, but not the superlative skills—at least that has been its uniform avowed purpose."

4. THE 2nd POINT OF ERROR: "SALZBERG INDICATES THE EQUIVALENCY OF THE CALCIUM AND ZINC SALTS."

This holding is directly contrary to the facts. Salzberg did not say that the calcium and zinc salts are equivalents. He merely said that from the acid esters which he produced and which acid esters are incidentally different from the acid esters from which Asseff produced his salts, calcium and zinc salts might be formed. The evidence of record in the present case, that is, the tests made with regard to the disclosure of the McNab patent shows that whereas Asseff's zinc salt is oil soluble, the corresponding calcium salt is not oil soluble.

5. THE 3rd POINT OF ERROR: "THEIR PRODUCTION (ZINC DICAPRYL DI-THIOPHOSPHATE) AND USE WAS NO MORE THAN WOULD BE REASONABLY EXPECTED FROM ONE SKILLED IN THE ART AND DID NOT CONSTITUTE INVENTION."

This Court in the case of *In re EK,* 57 App. D. C. 203, 19 F. (2d) 677 (1927), while considering the anticipatory effect of a foreign patent made the following observation which is applicable to the anticipatory effect of domestic patents as well. The court said,

> "A patent is to be measured as anticipatory, not by what might have been made out of it but by what is clearly and definitely expressed in it."

The Circuit Court of Appeals for the Third Circuit in the case of *Skelly Oil Co. v. Universal Oil Products Co.,* 31 F. (2d) 427 (CCA 3rd 1929), said,

> "A patent relied upon as an anticipation must itself speak. Its specification must give in substance the

> same knowledge and the same directions as the specification of the patent in suit. Otto v. Linford, 46 L. T. (N. S.) 35, 44. It is not enough to prove that a method or apparatus described in an earlier specification can be made to produce this or that result. Flour Oxidizing Co. v. Carr & Co., 35 R. P. C. 457. A singularly sensible test of the rule of anticipation is given in British Thomson-Houston Co. v. Metropolitan Vickers Electrical Co., 45 R. P. C. 22, by asking the question—'Would a man who was grappling with the problem solved by the patent attacked, and having no knowledge of that patent, if he had had the alleged anticipation in his hand, have said: 'That gives me what I wish?' The Pope Alliance Corporation v. The Spanish River Pulp & Paper Mills, Ltd. (Privy Council Appeals No. 33 of 1928)'."

There is always the tendency to interpret prior art references in the light of the knowledge derived from the one whose invention or patent thereon is under consideration. On that point Judge Buffington of the Circuit Court of Appeals for the Third Circuit in the case of *Hartford-Empire Co. v. Hazel-Atlas Glass Co.,* 59 F. (2d) 399, 413, said,

> "And where an art, eager for relief, found in these moribund patents nothing to meet that suggested solution, it is safer to rely evidentially on the then judgment, attitude, and conduct of the glass trade rather than on the post litem testimony of experts, the contentions of infringers, and the theoretical construction that often tempts courts to create out of lifeless patents an imaginary machine on paper which a working art could not do in steel."

Along the same line and with even greater clarity Judge Learned Hand in the case of *United Chromium Inc. v. International Silver Co.,* 60 F. (2d) 913 (CCA 2nd 1932), said,

> "We cannot safely say of retrospect that that was simple which skilled and ingenious experimenters did not contrive."

We must not forget that in the present case we have fortunately the record of what the skilled worker in the art was thinking at the time Asseff made his invention. We have that record in the abandoned application of Cook and Thomas. In their application they show clearly the "state of the art" at that time. Their statements show conclusively that the workers in the art were groping in the dark. They had no empirical rules to follow and in many instances a pure guess was ventured and it was found to be in error. A notable example of such a bad guess is their statement that the combination of a dithiophosphate and a detergent was inoperative. As stated by Judge Learned Hand in the *United Chromium* case,

> "Unless, therefore, the art had already learned as much empirically, his idea, when verified, has every mark of an invention."

Those very words apply to the Asseff invention.

6. THE 4th POINT OF ERROR: "ASSEFF WAS FOLLOWING * * * PRIOR ART WHEN HE ADDED A SEPARATE DETERGENT TO (ZINC) DICAPRYL DI-THIOPHOSPHATE SALT."

That this holding is clearly wrong is shown conclusively by reference to the abandoned Cook and Thomas application which when speaking of the possibility of the combined use of a detergent and a dithiophosphate these skilled workers in the art made the following guess:

> "It is interesting to compare the results obtained by the incorporation of small amounts of the heavy metal salts of dithiophosphoric acid esters in crankcase oils with the action of mixtures of dissimilar chemicals that have been added to oils in an attempt to accomplish the same purpose. Detergents such as calcium stearate, calcium naphthenate and the like have previously been used in admixture with anti-corrosion agents such as triphenyl phosphite and sulfurized sperm oil that have no detergent properties. Although

> each of these two classes of materials will perform its function in a lubricating oil the two separate chemicals do not cooperate to produce a satisfactory anti-corrosion and detergent action when used together. The function of a corrosion inhibitor is to cover the bearings and other corrodable metal surfaces of the automobile engine with a passivating film that prevents corrosion of the metal by the organic acids and other corrosive oxidation products in the oil. On the other hand a sludge inhibitor or detergent, as its name implies, operates to remove adhering solids from the metal parts of the engine and thus to produce a clean metal surface. *Consequently, the use of a corrosion inhibitor in admixture with the detergents heretofore employed has proven to be ineffective over any extensive period of time, as each chemical tends to offset the action of the other.*" (Emphasis ours.)

The foregoing is what the workers in the art thought at the time Asseff made his invention. How can it now be said that Asseff was following the prior art?

7. THE 5th POINT OF ERROR: "LEGALLY * * * THE EFFECTIVE DATE OF THE COOK PATENT (2,344,392) IS JULY 11, 1941, SINCE THE TWO APPLICATIONS (SERIAL NO. 401,960 FILED JULY 11, 1941, AND SERIAL NO. 418,326 FILED NOVEMBER 8, 1941, AND MATURED INTO PATENT 2,344,392) COVER THE SAME SUBJECT MATTER."

This is so obviously in error that no cases need be cited to show the error. A parent application is a basis for only what it discloses. The parent Cook application is defective in two respects. It does not show the dithiophosphate of Asseff's claims 1 and 5 and as to the remaining claims the parent case says that that combination is inoperative.

8. THE 6th POINT OF ERROR: "WILSON PATENT TEACHES THOSE SKILLED IN THE ART TO UTILIZE A PLURALITY OF ADDITIVE AGENTS AND THE PARENT COOK, ET AL. APPLICATION DISCLOSED THAT THE SPECIFIC HEAVY METAL SALTS OF THE DITHIOPHOSPHORIC ESTERS DISCLOSED THEREIN SHOULD BE USED WITH OTHER MATERIALS. THERE IS, THEREFORE, NOTHING INVENTIVE IN THESE CLAIMS."

The Wilson patent is concerned with a plurality of addition agents. But it is believed preposterous to hold that merely because Wilson put two things together and found them operative that any other two chemicals which one might select and use in a lubricant would likewise be compatible and operative. Here again we must revert to the statement contained in the abandoned Cook and Thomas application which was made at a date subsequent to the filing of the Wilson application and which said that the combination in question was believed to be inoperative.

9. THE 7th POINT OF ERROR: "THE INCLUSION OF THIS MATERIAL (PHENATES), OLD IN THE ART, WITH THE BASIC COMPOUND OF THE COOK PATENT, IS NOT NEW OR INVENTIVE."

This statement is likewise refuted by what Cook and Thomas said in their abandoned application. They were speaking of dithiophosphates generally which they in that application intended to use alone. They did not disclose the zinc dithiophosphate of the present claims. If their statement was not a pure guess then it was based on tests, not reported, but with other materials of unknown composition which gave them the failures. Whatever may have been the basis for the statement contained in the abandoned application it must be recognized that such statement is a logical one based on what might be *expected* to happen when a detergent and a corrosion inhibitor such as a dithiophosphate were used in combination. The expected results as stated by Cook and Thomas would be a failure because "each chemical tends to offset the action of the other." Yet it is this combination which when made and tested by Asseff was shown to have an overall effectiveness which was greater than the mere cumulative effect of the two materials when used alone. The result discovered by Asseff was therefore unexpected in two respects. First, the two addition agents did not counteract each other but second, they cooperated in providing a detergency effect which was greater than could be expected by the use of the detergent material by itself.

10. THE 8th POINT OF ERROR: "PLAINTIFF'S CLAIMS FAIL TO ANTE-DATE THE ANTICIPATORY EFFECT OF THE COOK ET AL. PATENT."

It has been fully demonstrated that the effective date of the Cook patent, insofar as the claims in issue are concerned, is the filing date of the application on which it is based and *not* the parent case. For the reasons previously pointed out the disclosure of the Cook et al. applications would lead one *away* from the use of the zinc salt and their parent case specifically stated that the combination of a dithiophosphate with any detergent was not operative.

In the application which Cook and Thomas filed on July 11, 1941, they were concerned with improving lubricants by the addition thereto of heavy metal salts of dithiophosphoric acid esters. The only tests on which data were reported by Cook and Thomas in their July 11 application are set forth in Tables I and II, pages 171A and 172A. These tests were merely laboratory oxidation tests. The two samples in Table I were both barium salts, and of the eight samples in Table II, six were barium salts and two were zinc salts. With regard to the zinc salts, no data is given in Table II on anything but corrosion. The two compounds which can be directly compared in Table II are as previously indicated, the third and last compounds in that table, which differ from each other only in the metal employed. In the third compound the metal was barium, and in the last compound the metal was zinc. These data show that whereas the barium material was definitely a corrosion inhibitor, the corresponding zinc salt was decidedly corrosive. On page 165A, at line 20, Cook and Thomas give as their preferred example the barium salt. Again on page 168A, in line 25, their invention is expressed as comprising the use of "barium and other heavy metal salts." In the first nine lines on page 169A, Cook and Thomas first list "zinc, cadmium, aluminum, tin and lead" together as comprising a group of metals which might be used. A preference is indicated for "magnesium, calcium, barium, and strontium." On page 169A, beginning with line 12, the "alkaline earth metal salts" are indicated as "particularly advantageous * * * because they are quite soluble in hydrocarbon oils." This is a striking example of the fact that Cook and Thomas were guessing. In the statement last quoted they would lead one to believe that the calcium salts of all of the dithiophosphoric acids are preferred because they are outstandingly oil-soluble. The actual fact is quite the opposite, as shown in Exhibit 24 in which test it was demonstrated conclusively that both McNab, and Cook and Thomas were guessing, and guessing wrongly about this particular type of material.

On page 170A, beginning with line 5, Cook and Thomas said that "the barium, calcium, and other alkaline earth metal salts of di-octyl and di-lauryl dithiophosphate, for example, are soluble in ordinary 10-W grade lubricating oil to the extent of 50% to 80% at 25°C." No data whatsoever are given in support of that statement, which has now been shown to be erroneous.

The point of the foregoing is that absolutely all that Cook and Thomas knew at the time they filed their July 11, 1941 application was the data given in Tables I and II of their application, pages 171A and 172A. Their evaluation of the zinc compounds as being inferior is borne out by the fact that whereas they gave data with regard to A. P. I. gravity; naphtha insolubles and Neut. No. with regard to all of the barium compounds tested, no such data were given with regard to the zinc compounds. Either they did not believe them of sufficient importance in view of their inferior behavior on corrosion, or else the results were so damaging that such results were purposely omitted.

Then on October 28, 1941, Asseff's patent 2,261,047 was issued. Asseff, in that patent gave data from the results of 150 hour engine tests on eleven different lubricant compositions. The Asseff patent, therefore, gave real information. Its disclosure was not based on mere beaker tests. He subjected his lubricants to the type of engine test which is recognized by the industry as a proper evaluation of a lubricant of this character. All of those data in the Asseff patent, you can be sure, were very carefully analyzed and studied by Cook and Thomas, who are employed in the laboratory of a company which is competitive with the company in whose laboratory Asseff did his work. Cook and Thomas then on November 8, 1941 filed their application, which matured into patent 2,344,392. If we compare the specification of the application which Cook and Thomas filed on November 8, 1941 with the specification of their application filed on July 11, 1941, we find the following striking differences. First, they now say that the dithiophosphates can be used with detergents. No basis, from the standpoint of technical data, was given by Cook and Thomas in their November 8th application for this complete reversal of position. Their November 8th specification contains no data whatsoever, with the exception of a laboratory oxidation test run on three barium compounds. Not a single bit of information with regard to any engine runs was given by them. They did not even run an oxidation test on the combination of the thiophosphate and a detergent.

The second principal difference between the July and November applications of Cook and Thomas is that the November case made brief reference to an alleged preparation of zinc dicapryl dithiophosphate, although here again no details of such preparation are given, and no data whatsoever are given with regard to the performance of that material.

A further marked difference between the two applications is the note which appears on page 183A, first column, line 27, to the effect that whereas the alkaline earth metal salts are brownish-yellow solids, the zinc salt is a viscous liquid at ordinary temperatures.

All of these changes were made by Cook and Thomas in filing their November 8, 1941 application. The changes thus made are all directly attributable to the Asseff patent disclosure. There are no data in the November 8, 1941 Cook and Thomas application which can be relied upon as the basis for Cook and Thomas having so radically changed their minds since the filing of their previous application in July. This much can be said for Cook and Thomas, however, they did not claim any of the subject matter of the present invention. They did not claim, in their November 8, 1941 application, what they had learned from the Asseff patent. They were not willing to make oath to the fact that what they had learned from the Asseff patent was their invention. The only thing claimed by them as their invention in the November application, was the use in lubricants of the heavy metal salts of dicapryl esters of dithiophosphoric acid. The foregoing demonstrates conclusively that Asseff is the prior inventor of the claims here in issue. It demonstrates clearly that any statement found in the second Cook and Thomas application which has any bearing whatsoever on the invention of the claims here at issue would appear to have been derived by them from a reading of Asseff's patent.

11. THE 9th POINT OF ERROR: "PLAINTIFF'S CLAIMS 9 THROUGH 24 LACK INVENTION AND PATENTABILITY OVER McNAB, SALZBERG AND WILSON."

This seems to be a sort of catch-all blanket rejection and nowhere in the trial court's decision is there found any basis for this statement other than the statements which were made by the trial court in support of the previously enumerated points of error. Since these patents singly and in combination have been analyzed in great detail in the preceding portions of this brief and since we have clearly shown wherein it is not possible to spell out of any of them or any combinations of them even a suggestion of Asseff's invention, it is believed that this particular point of error need not be elaborated upon.

VI.

SUMMARY.

1. The claimed invention of Asseff is the specific discovery that the *zinc* salt of the acidic reaction product of *capryl* alcohol with P_2S_5 gives a totally unexpected improvement.

2. That improvement is specific to the specific material claimed.

3. That improvement is not only maintained but actually enhanced by the additional presence of the specifically claimed cooperating addition agents.

4. Asseff was the first to ever make and test in an oil the claimed zinc salt.

5. Nothing in the prior art pointed toward this specific material:

   (a) Salzberg's work would lead one to try long chain rather than short chain alcohols.

   (b) Wilson's work would lead one to try other weak acids rather than the much more strongly acidic material produced when reacting capryl alcohol with P_2S_5. Moreover Wilson discloses no thiophosphates.

(c) McNab's work would lead one to try various mixtures of various metals with zinc not included in the list from which the metals would be selected.

6. The contemporary work of Cook and Thomas led them away from the zinc compound, they having selected the barium compound as being best.

VII.

RELIEF.

For the reasons hereinbefore stated, the judgment of the District Court should be reversed and it should be decreed that plaintiff Asseff is entitled to receive a patent on the claims in issue.

Respectfully submitted,

Oscar C. Limbach,
Leader Bldg.,
Cleveland, Ohio,
Attorney for Plaintiffs-Appellants.

Almon S. Nelson,
604 Warner Bldg.,
Washington, D. C.,
Of Counsel.

BRIEF FOR APPELLEE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA CIRCUIT

APPEAL No. 10,520

PETER A. ASSEFF AND THE LUBRI-ZOL CORPORATION,
APPELLANTS

v.

JOHN A. MARZALL, COMMISSIONER OF PATENTS,
APPELLEE

APPEAL FROM THE JUDGMENT OF THE UNITED STATES DISTRICT COURT FOR THE DISTRICT OF COLUMBIA CIRCUIT

E. L. REYNOLDS,
Solicitor, United States Patent Office,
Attorney for Appellee.

J. SCHIMMEL,
Of Counsel.

United States Court of Appeals
for the
District of Columbia Circuit

FILED APR 14 1950

Joseph W. Stewart
CLERK

BRIEF FOR APPELLEE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA CIRCUIT

APPEAL NO. 10,520

PETER A. ASSEFF AND THE LUBRI-ZOL CORPORATION,
APPELLANTS

v.

JOHN A. MARZALL, COMMISSIONER OF PATENTS,
APPELLEE

APPEAL FROM THE JUDGMENT OF THE UNITED STATES DISTRICT COURT FOR THE DISTRICT OF COLUMBIA CIRCUIT

E. L. REYNOLDS,
Solicitor, United States Patent Office,
Attorney for Appellee.

J. SCHIMMEL,
Of Counsel.

United States Court of Appeals
For the
District of Columbia Circuit

FILED APR 14 1950

Joseph W. Stewart
CLERK

INDEX

Page
Introduction........ 1
Appellants' application........ 2
The claims........ 3
The prior art........ 4
Summary of rejections........ 6
Summary of argument........ 7
The argument........ 8
  1. The Cook et al. patent........ 8
  2. Other prior art........ 18
  3. Indefiniteness........ 20
Conclusion........ 24
Authorities Cited:
  Abbott v. *Coe*, 71 App. D. C. 195, 109 F. 2d 449........ 24
  Alexander Milburn Co. v. *Davis etc.*, 270 U. S. 390........ 9
  American Lecithin Co. v. *Warfield*, 7th Cir., 128 F. 2d 522........ 11
  Asseff v. *Kingsland*, 178 F. 2d 710........ 17
  Barber v. *Otis Motor Sales*, 2d Cir., 271 Fed. 171........ 11
  Brush v. *Lexington*, 14 F. 2d 108........ 22
  Crampton Mfg. Co. v. *Crampton*, 68 USPQ 122........ 22
  Deering v. *Winona Harvester Wks*, 155 U. S. 286........ 11
  Ellis v. *Shaw*, 54 App. D. C. 185, 205 Fed. 1006........ 16
  Godfrey v. *Eames*, 68 U. S. 317........ 13
  Graver Tank et al. v. *Linde Air Products*, 80 USPQ 451........ 23
  Holland Furn. Co. v. *Perkins Glue Co.*, 277 U. S. 245........ 21
  Hunt v. *Mallinckrodt Chem. Wks*, 72 F. S. 865, 74 USPQ 122........ 13
  Hunter v. *Stikeman*, 13 App. D. C. 226........ 11
  Knutson et al v. *Gallsworthy*, 82 U. S. App. D. C. 304........ 12
  Lotz, In re, 57 App. D. C. 402........ 13
  Mathieson Alkali Wks. Inc. v. *Crowely*, 78 U. S. App. D. C. 163, 138 F. 2d 281........ 11
  Miessner Inc. v. *Hosehke et al*, 76 U. S. App. D. C. 343........ 12
  Minnesota Mining & Mfg. Co., v. *Coe*, 69 App. D. C. 256, 100 F. 2d 429........ 9, 16
  Murphy & Chadeloid Chemical Co. v. *Coe*, 69 App. D. C. 225........ 23
  Oliver Mch. Co. v. *Gellman*, 6th Cir., 104 F. 2d 11........ 11
  Pearce v. *Mulford*, 102 U. S. 112........ 16
  Petrie v. *DeSchweintz*, 19 App. D. C. 386........ 11
  Radtke et al. v. *Coe*, 74 App. D. C. 251, 122 F. 2d 937........ 24
  Rembert et al v. *Coe*, 78 U. S. App. D. C. 51........ 19
  Smith v. *Goodyear Dental Vulcanite Co.*, 93 U. S. 486........ 13
  Sonneborn Sons Inc. v. *Coe*, 70 App. D. C. 97........ 17
  Standard Brands v. *National Yeast*, 308 U. S. 304........ 22
  Standard Oil v. *Marzall*, 84 USPQ 363........ 24
  Thompson v. *American Tobacco Co.*, 4th Cir., 174 F. 2d 773........ 11
  Townsend v. *Smith*, 17 C. C. P. A. 647; 36 F. 2d 292........ 11
  U. S. v. *Dubilier Condenser Corp.*, 298 U. S. 178........ 11

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA CIRCUIT

APPEAL No. 10,520

PETER A. ASSEFF AND THE LUBRI-ZOL CORPORATION,
APPELLANTS

v.

JOHN A. MARZALL, COMMISSIONER OF PATENTS,
APPELLEE

APPEAL FROM THE JUDGMENT OF THE UNITED STATES DISTRICT COURT FOR THE DISTRICT OF COLUMBIA CIRCUIT

BRIEF FOR THE COMMISSIONER OF PATENTS

INTRODUCTION

This is an appeal from the judgment (App. 22) of the United States District Court for the District of Columbia, dated October 17, 1949, dismissing appellants' complaint (App. 2–9) against the Commissioner of Patents in an action under Section 4915 of the Revised Statutes (35 U. S. C. sec. 63), Civil Action 3967–47, in which appellants sought to have the District Court issue an order authorizing the Commissioner to grant a patent containing certain claims, numbered 1, 5 and 9 to 24 inclusive (App. 3–7), of the Asseff patent application, Serial No. 457,746, filed September 9, 1942, on an alleged invention in a "Lubricating Composition." The complaint was dismissed on the grounds that the findings of the Court established that the Cook et al. patent anticipates all the claims, and that claims 9 through 24 lack invention over the patents to McNab, Salzberg and Wilson (App. 21, 22).

No separate findings of fact or conclusions of law were made, since the memorandum opinion of the Court (App. 13–22) was stated to include such findings (App. 22).

APPELLANTS' APPLICATION

The specification of the application at bar (App. 114–128) describes the invention as one relating to lubricating compositions which are particularly suited for use under high temperature conditions such as those encountered in internal combustion engines of the Diesel type. The main or base constituent of the composition is a hydrocarbon oil such as mineral lubricating oil, and in which the minor constituent is a compound, the molecular structure of which includes the combination of zinc, the capryl alcohol radical and the dithiophosphate radical. Other compounds may be included in the composition such as detergents, pour point depressors and extreme pressure agents to meet particular problems of lubrication (Memo. opinion of the trial court App. 13, 14).

The compound including in its structure the combination of zinc, the capryl alcohol radical and the dithiophosphate radical is denominated the characterizing addition agent of appellants' composition (App. 118), and is defined in all the claims as "the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulfide" (App. 15).

THE CLAIMS

The alleged invention is defined by the claims appearing in the complaint (App. 3–7). Although all the claims are in composition form they fall into four groups as follows:

1. Claims 1 and 5 (App. 3) which call for a single addition agent, the zinc salt of dicapryl dithiophosphate;

2. Claims 9 to 14 inclusive (App. 4, 5) and 22 to 24 inclusive (App. 7) which are limited to the use of a second addition agent in the composition, defined broadly as a "detergent of the class consisting of oil soluble organic compound containing a metal atom directly attached to an element of the class consisting of oxygen and sulfur" in claims 9, 10, 12, and 13; defined more specifically as "a calcium salt of a petroleum sulfonic acid" in claims 11 and 14, and as an oil soluble "metallic phenate" in claim 22, which is still more specifically defined as a metallic phenate derived from an ester of "salicylic acid" in claim 23, and as "the magnesium phenate derived from lauryl salicylate" in claim 24;

3. Claims 15 and 16 (App. 5) which also require a second addition agent, which is defined broadly in claim 16 as an "oil soluble organic corrosion inhibitor," and more specifically as "a halogen bearing organic compound having a vapor pressure less than atmospheric at 140° C." in claim 15; and

4. Claims 17 to 21 inclusive (App. 5, 6) which require the presence in the composition of three addition agents, namely the zinc salt of capryl dithiophosphate, the detergent and the corrosion inhibitor. In these claims the detergent is defined broadly as in claims 9, 10, 12, and 13, while the corrosion inhibitor is defined broadly in claims 18 and 20, and more specifically, as in claim 15 in claims 17, 19, and 21.

As pointed out in the memorandum opinion of the trial court "all the claims are distinguished by the inclusion therein of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulfide, and it is this product upon which the plaintiffs base their contention of patentable novelty in the claims" (App. 14).

THE PRIOR ART

On December 8, 1936, a patent, 2,063,629, (App. 190–193) was granted to Salzberg et al. for a compound employed as a corrosion inhibitor in lubricants for bearings which operate under high pressure (p. 3, col. 2, lines 41–43). The patent relates to the production of acid esters and salts of dithiophosphorus acid. Zinc is mentioned as one of the metals forming the salt (p. 3, col. 2, line 19) and the ester portion is provided by aliphatic alcohols containing at least ten carbon atoms (p. 1, col. 1, lines 33–36) (App. 15).

On July 14, 1942, a patent, 2,289,795 (App. 185–189) was issued to McNab, on an application filed September 1, 1939 for compositions related to improved lubricants and especially crank case oils for the lubrication of internal combustion engines and particularly for high pressure fuel injection engines such as Diesel engines. The patent discloses the use of a salt of a dithiophosphoric acid ester, especially calcium di-octyl dithiophosphate (p. 3, col. 2, line 70) in conjunction with metal soaps of natural and synthetic fatty acids and naphthenic acid (p. 2, col. 2, lines 56–61). The McNab patent teaches that viscosity improving agents and pour point depressors may also be included for their particular function (p. 4, col. 2, lines 17–30) (App. 15).

On April 21, 1942, a patent, 2,280,419 (App. 194–200) was granted to Wilson, on an application filed July 16, 1940, which patent describes a lubricant containing an oil soluble salt of the so-called "mahogany" sulfonic acids, including the zinc salt, together with oil soluble metal salts of thiophenols (p. 1, col. 1, lines 37–45). The sulfonic acids function as detergents, while the sulfur of the thiophenol imparts anticorrosive properties (p. 4, col. 1, lines 35–43) (App. 16).

On March 14, 1944, a patent, 2,344,392 (App. 183, 184) was granted to Cook et al., on an application filed November 8, 1941, as a continuation in part of an application filed July 11, 1941. This patent describes the preparation of heavy metal salts of the dicapryl ester of dithiophosphoric acid, and teaches that such salts are useful as sludge preventers and anti-corrosive agents for lubricating oils used in internal combustion engines (p. 1, col. 1, lines 8–11 and 42–47). Zinc is specifically mentioned as one of the heavy metals (p. 1, col. 1, lines 1–23). The heavy metal salts are stated to cooperate with heavy metal salts of diamyl phenol sulfides described in the Cook et al. patent 2,299,626, as well as with alkaline earth metal salts of petroleum sulfonates (p. 1, col. 2, lines 48–53). The dithiophosphoric acid ester salts are described as being good detergents and to possess important non-oxidizing or anti-oxidant properties (p. 1, col. 1, lines 43–48), and as being usable jointly with other detergents and corrosion inhibitors (App. 16).

On October 8, 1941, a patent, 2,261,047, (App. 177–182) was granted to Asseff, on an application filed July 28, 1941, covering a compounded lubricant. The patent describes a zinc methyl cyclohexanol dithiophosphate salt as an additive for lubricating oil and discloses that metal phenates may be used in conjunction therewith (p. 2, col. 1, lines 6–12) (App. 16).

SUMMARY OF REJECTIONS

In the Patent Office the appealed claims were refused by the Board of Appeals because unpatentable over the patent to Cook et al. and over McNab or Wilson in view of Salzberg et al. In addition claims 16, 18, 20, and 21 were held to be too broad and indefinite with respect to "corrosion inhibitors" (App. 176).

Defendant's answer (App. 12) denied that the claims were patentable both because of the prior art relied upon by the examiner and the Board of Appeals, and because the claims are indefinite in that they fail to point out and distinctly claim the part, improvement of combination as required by Sec. 4888 R. S. (35 U. S. C. 33).

At the trial appellants' counsel conceded that the Cook et al. patent does contain a disclosure of the subject matter of the appealed claims, but stated that the evidence to be introduced would show that the plaintiff Asseff made his invention prior to the effective date of that patent (App. 28).

In the trial court it was found

> (1) that plaintiffs' claims fail to antedate the anticipatory effect of the Cook et al. patent, which anticipates all of the claims; and
>
> (2) that plaintiffs' claims 9 through 24 lack invention and patentability over McNab, Salzberg and Wilson (App. 22).

On the question of indefiniteness, the court stated that "it is unnecessary for the Court to pass upon the defendant's contention concerning the plaintiff's failure to meet statutory requirements relating to particularity and distinctness" (App. 22).

Should this Court sustain findings (1) and (2) above of the District Court, it would be evident that no ruling need be made upon the question of indefiniteness, but in the event that this Court should not approve those findings, it is believed to be necessary to present herein the reasons why the Patent Office considers that the claims fail to meet the requirements of R. S. 4888.

SUMMARY OF ARGUMENT

1. The Cook et al. patent is prior art against appellants, both because the evidence submitted to antedate the anticipatory effect of the patent is inadequate

and insufficient, and because the effective date of that patent is the filing date of the parent application of which the patent is a continuation-in-part.

2. Since Cook et al. patent is prior art and since it discloses the essential feature of appellants' alleged invention, subsidiary features being old in the Wilson patent and the McNab patent, the concept of adding conventional additives to the basic composition disclosed in Cook et al. is obvious.

3. The McNab patent discloses the conjoint use of a soap detergent and a corrosion inhibitor of the class used by appellants, while the Salzberg patent discloses appellants' specific corrosion inhibitor.

4. The concept of substituting Salzberg's corrosion inhibitor for McNab's corrosion inhibitor is obvious and involves no more than routine experimentation.

5. Certain claims are indefinite because incomplete and because of expressions such as "corrosion inhibitor" and "characterizing amount."

THE ARGUMENT

1. The Cook et al. patent

Since the record shows that appellants admit that the Cook et al. patent does disclose the use in a lubricant of the zinc salt of the reaction product of capryl alcohol and phosphorus pentasulfide (App. 28 and Br–12), and since appellants' sole contention with regard to this patent is that Asseff made his invention as of a date prior to the invention date of Cook (Br–12), the issue as to all claims revolves around this alleged antedating evidence.

Claim 1 sets forth a lubricating composition containing a characterizing amount of the product obtained by the *simultaneous admixture* of capryl alcohol, phosphorus pentasulfide and zinc oxide. Examination of the evidence submitted by appellants as it relates to the limitations of this claim show that the inventor testified that "no exhibit offered related to any simultaneous admixture" of the reacting materials (App. 68). Accordingly, under these circumstances, appellants are remitted to the filing date of the Asseff application, September 9, 1942, which date being subsequent to the filing date of the Cook et al. patent, November 8, 1941, conclusively establishes the Cook et al. patent as prior art against this claim. (*Alexander Milburn Co.* v. *Davis etc Co.* 270 U. S. 390; *Minnesota Mining & Mfg. Co.* v. *Coe* 69 App. D. C. 256, 100 F (2d) 429.) It would appear significant that appellants' brief is silent as to this limitation of claim 1. Though the Cook et al. patent discloses the preparation of the reaction product by adding zinc oxide to a solution of dicapryl dithiophosphoric acid (App. 183–p. 2, col. 1, lines 38–45), the product is essentially the same, whether the reactants are added sequentially or simultaneously. Appellants so state (App. 118). However, because the claim is limited to "simultaneous admixture," and because no proof of the prior inventorship of the claimed subject matter was presented, it is submitted that the finding of the trial court that this claim is anticipated by Cook et al. (App. 18) was proper and should be affirmed.

Claim 5 likewise is a single additive claim defining broadly the combination of a mineral oil and a minor amount of the product obtained by preparing the zinc salt of the reaction product of capryl alcohol with phosphorus pentasulfide. The inventor Asseff in his testimony identified two exhibits, 23 and 24, examples 6 and 7 code Nos. 601–302 and 601–310 (App. 58 and 60) as establishing prior invention of the composition defined in this claim. Appellants appear to rely primarily upon example 6 (Br. 20) and urge that the exhibit shows completion of the invention on October 3, 1941 (Br. 14).

With respect to appellants' exhibit 23, example 6, code No. 601–302 (App. 58), it should be noted that the zinc reaction product was stated to have been first prepared on September 11, 1941, tests started September 25, 1941, which were completed on October 3, 1941 (App. 66). However, it is important to note that the inventor Asseff did not make the compound, did not test the composition containing the same, and did not make the note-book entries relating thereto (App. 49, 50, 68). No evidence was submitted to prove those entries by the party making them and no evidence was introduced to show that the party who did the actual work did so at the suggestion of the party Asseff. Most of the other exhibits presented in evidence, entries from notebooks of the party Asseff do not relate to the specific subject matter which vitalizes the claims sought here. Though that evidence may show extensive experimentation with metal salts of esters of dithiophosphoric acid generally there is nothing therein pointing to the zinc salt of the dicapryl dithiophosphate as under investigation or consideration. Dr. Smith corroborated that the entries were made as testified to by Asseff (App. 75, 76). Dr. Smith's testimony on the question of prior reduction to practice is meager and vague (App. 76).

The law is well settled that where one seeks to carry his date of invention back of the date of an anticipating patent, he assumes the burden of proof and must establish his claim by evidence so clear, unequivocal and cogent as to leave no reasonable doubt. (*Deering v. Winona Harvester Works,* 155 U. S. 286; *Barber* v. *Otis Motor Sales Co.,* 2nd Cir., 271 F. (2d) 171; *Oliver Machinery Co.* v. *Gellman,* 6th Cir., 104 F. (2d) 11; *American Lecithin Co.* v. *Warfield Co.,* 7th Cir., 128 F., (2d) 522; *Thompson* v. *American Tobacco Co.,* 4th Cir., 174 F. (2d) 773. Likewise, the law is well settled that invention is the result of an inventive act; it consists in conceiving an idea and reducing it to practice. (*U. S.* v. *Dubilier Condenser Corp.,* 289 U. S. 178). Conception and reduction to practice must be clearly established by proof (*Townsend* v. *Smith,* 17 C. C. P. A. 647, 36 F. (2d) 292;) and reduction to practice must be shown to have been performed by the inventor or his agent at his instance (*Hunter* v. *Stickeman,* 13 App. D. C. 226). Personal testimony of the inventor must be corroborated (*Mathieson Alkali Works Inc.* v. *Crowley,* 78 U. S. App. D. C. 163, 138 F. (2d) 281).

Since the established rule is that there must be independent corroboration of all the essential factors concerned in reduction to practice (*Petrie* v. *De*

Schweinitz, 19 App. D. C. 386) (*Miessner Investment* v. *Hosehke et al.,* 76 U. S. App. D. C. 343) (*Mathieson Alkali* v. *Crowley*), *supra*—it is submitted that appellants' contention that the party Asseff reduced the claimed subject matter to practice prior to the filing date of the Cook et al. patent stands uncorroborated in the record. Neither Asseff or Smith testified that they saw the specific compound prepared or that they were present at the time the lubricating compositions were compounded and tested. Under these circumstances, it is submitted that the evidence relied upon to establish an earlier reduction to practice is hearsay, inconclusive, inadequate and insufficient. It does not meet the test laid down by the authorities cited above, that the evidence must be so cogent as to leave no reasonable doubt that the anticipating patent is antedated. It is submitted that appellants' contention that Asseff made the claimed invention as of a date prior to the filing date of the Cook et al. patent has not been proved.

With respect to appellants' exhibit No. 24, example 7, code No. 601–310 (App. 60), it appears clearly from the testimony that no test of that composition was made, only preparation of the reaction product and storage thereof is testified to (App. 61). The mere preparation of that composition, it is submitted, is not sufficient as a reduction to practice. Testing and successful operation under normal operating conditions of lubricating compositions are necessary to establish reduction to practice (*Knutson et al.* v. *Gallsworthy,* 82 U. S. App. D. C. 304, 164 F. (2d) 497). Accordingly, in legal contemplation, the mere dissolution of the zinc salt described in the testimony of the witness, without more, cannot be a reduction to practice of the composition of claim 5. Moreover, as pointed out in the opinion of the court below the preparation of the composition was more than two months subsequent to the filing date of the parent Cook et al. application, and accordingly, covered by the prior disclosure of Cook et al. (App. 20).

However, even assuming, arguendo, that appellants have met the burden of proof and have established completion of the invention defined in claim 5 as of October 3, 1941, as contended (Br. 14), nevertheless it is submitted that the Cook et al. patent has not been eliminated as prior art. This patent states on its face that it is a continuation in part of an application filed July 11, 1941 (App. 184, col. 2, lines 29–31). Legally the effective date of the Cook et al. patent is July 11, 1941 if the two applications cover the same subject matter (*Godfrey* v. *Eames,* 68 U. S. 317; *Smith* v. *Goodyear Dental Vulcanite Co.,* 93 U. S. 486;) and for all matter carried over into the continuing application from the parent application, the continuation in part application receives the benefit of the parent application's filing date. That is well settled in the law. (*Hunt* v. *Mallinckrodt Chemical Works,* 72 F. S. 865, 74 USPQ 122; *In re Lotz,* 57 App. D. C. 402).

Examination of the Cook et al. parent application (App. 164–172) shows that the efficacy of the oil soluble heavy metal salts of dithiophosphoric acid esters as detergents in lubricating oil compositions and as antioxidants was clearly recognized (App. 165). These salts were used alone or in admixture with other ingredients. Preference is indicated for the salt esters in which each alkyl group contains 8, 10, 12 or 14 carbon atoms (App. 168) and in which the heavy metal is zinc, cadium, aluminum, tin, lead and the alkaline earth metals which are magnesium, calcium, strontium and barium (App. 169). A dicapryl dithiophosphate salt is specifically disclosed in Table II (App. 172). The conjoint use of the dithiophosphate ester with dialkylphenol sulfides is also taught, the latter compound being used to put into solution larger quantities of the dithiophosphate ester (App. 168).

While the disclosure of the Cook et al. parent application is generic to alkyl groups from 8 to 14 carbon atoms, and the Cook et al. patent is specific to the alkyl group being a capryl radical, it is to be noted that the capryl group is but a species of the alkyl groups containing 8 carbon atoms, and since the use of that specific alkyl radical was disclosed in the early Cook et al. application, it is submitted that that disclosure is prior art insofar as appellants are concerned. Moreover, since the disclosure of the heavy metals of the parent application is carried over into the patent without any essential change, that disclosure is also prior to appellants. Although it is conceded that the early Cook et al. application did not disclose the preparation of a zinc dicapryl dithiophosphate salt, as appellants contend (Br. 14), it did disclose all the components of the reaction product defined in the claim even to the preference for zinc as the heavy metal, and for octyl and capryl groups as the alkyl radical. Since methods for the production of the heavy metal salts of esters of dithiophosphoric acid were known to the art, and appellants' witness, Dr. Smith admitted that the ordinary chemist would experience no difficulty in making the zinc salt of dicapryl dithiophosphate (App. 98), it is submitted that the parent Cook et al. application is prior art against appellants and that this claim is unpatentable over the Cook et al. patent.

Claims 9, 10, 12, and 13 within the second group of claims listed above define compositions containing a second additive, said to be a detergent, and described as a compound containing a metal atom directly attached to an element of the class consisting of oxygen and sulfur. Cook et al. discloses in the parent application that the dithiophosphate salt ester may be used in admixture with other additives (App. 165), and the metal salts of dialkylphenol sulfides specifically mentioned (App. 168) respond fully to the definition of the second additive of this claim. Appellants' contention that the earliest disclosure of any combination of a thio-phosphate with any other additive by Cook et al. occurred on November 8, 1941 when the continuing application was filed (Br. 15) is, therefore, clearly without merit. Since the compound fully meets the definition thereof set forth in the claim it is immaterial that the prior disclosure fails to designate its function or property of detergency since that function is obviously an inherent one, and appellants are not entitled to a patent for the mere statement of an inherent feature (*Ellis* v. *Shaw,* 54 App. D. C. 185, 205 Fed. 1006).

Claims 11 and 14 are for the same combination as claim 9, but define the second additive more specifically as "a calcium salt of a petroleum sulfonic acid." The use of those specific materials as detergents was known to the art prior to either Cook et al. or the appellants, as clearly shown by the patent to Wilson (App. 194). Since Cook et al. in the early application clearly disclosed that other additives could be used with the dithiophosphate salt esters, it is submitted that appellants were only following in the path clearly outlined by the prior art in adding the Wilson detergent to the composition; and even conceding or assuming that such addition was an improvement, improvement does not necessarily connote invention, particularly where all that has been done is well within the skill of the art. (*Pearce* v. *Mulford,* 102 U. S. 112; *Minnesota Mining and Mfg. Co.* v. *Coe,* 69 App. D. C. 256.)

Claims 22, 23, and 24, likewise, are for the same combination as claim 9, but define the second additive more specifically as a "metallic phenate." These compounds were known to the art as detergents long before either Cook et al. or appellants filed, as shown by the patents to McNab (App. 185) and Wilson (App. 194). For the same reasons as those set forth in the preceding paragraph these claims, too, are submitted to be unpatentable over Cook et al.

Claims 15 and 16, listed in group 3 above, define a second addition agent, but as set forth in these claims the second agent is a corrosion inhibitor, instead of a detergent, and is defined as a "halogen bearing organic compound." Since the use of such compounds was old in the art, as shown by Wilson (App. 194), the addition of such a compound to the dithiophosphate ester salt of Cook et al. involves nothing inventive (*Asseff* v. *Kingsland,* 178 F. 2d 710).

Claims 17 to 21, inclusive, listed in group 4 above, define a composition containing the zinc dicapryl dithiophosphate combined with the detergent as defined in claim 9 and the corrosion inhibitor as defined in claims 15 and 16. As to these claims it is to be noted that the prior art as constituted by both Cook et al. and Wilson patent teach that a plurality of addition agents are more beneficial than a single one (App. 200, col. 1, lines 19–39). The latter in particular also shows the combined use of a detergent and corrosion inhibitor (page 4, col. 1, lines 27–42). In the light of these disclosures, it is submitted that the conjoint use of Wilson's detergent and corrosion inhibitor in Cook et al.'s basic composition would be obvious and well within the skill of the art. Moreover, mere routine experimentation of the prior art teachings with the newer additives constitutes no more than good engineering practice and not invention (*Sonneborn Sons Inc.* v. *Coe,* 70 App. D. C. 97).

Accordingly, it is submitted that claims 1, 5, 9, 10, 12, and 13 are fully anticipated by Cook et al.; that claims 11 and 14 to 24, inclusive, are unpatentable over Cook et al. in view of Wilson, and that claims 22 to 24, inclusive, are unpatentable also over Cook et al. in view of McNab.

2. Other prior art

Claims 9 to 24, inclusive, which define compositions containing plural additives were considered by the tribunal of the Patent Office and by the trial Court to be unpatentable over the disclosures of McNab, Wilson and Salzberg (App. 22).

As pointed out previously the prior art as represented by Wilson (App. 194) and McNab (App. 185) recognized that the use of a plurality of additives in a lubricating oil composition was advantageous and gave improved results. In addition, Wilson recommended that compounds containing halogen could be used with detergents for improved film strength (page 6, col. 1, line 1). McNab taught the art the use of metal soaps in conjunction with a salt of dioctyl dithiophosphate far prior to appellants' application for patent here in suit. Each of these patents teaches the conjoint use of a detergent and a corrosion inhibitor and further McNab indicates that other addition agents, such as thickeners, viscosity index improving agents, pour depressors, may be included for their particular function (App. 188, col. 2, lines 17-30).

Salzberg (App. 190) relates specifically to methods for producing acid esters and salt esters of dithiophosphoric acid, and indicates that such compounds act as corrosion inhibitors in lubricants for bearings (page 3, col. 2, lines 40-43). The disclosed invention embraced the acid esters and their salts such as the alkali, alkaline earth, calcium, zinc, lead and ammonium salts (page 3, col. 2, lines 17-19).

Accordingly, the prior art was conversant with the use of salts of dialkyl dithiophosphate, and specifically dioctyl dithiophosphate. Since the alcohol used by appellants in the preparation of the alkyl thiophosphate—capryl alcohol—is admittedly an aliphatic alcohol (App. 90) and an octyl alcohol (App. 88)—the use thereof falls clearly within the disclosure of the prior art. The preparation and use of the capryl ester was a logical and obvious adaptation of the teaching of this prior art, requiring for its accomplishment only the skill of the art. Moreover, Salzberg indicated that zinc and calcium salts would be operative as corrosion inhibitors, and McNab substantiates that observation by his specification of calcium dioctyl dithiophosphate in conjunction with other additives for the same purpose. The substitution of the zinc salt for the calcium salt in McNab was well within the skill of the art, and even if improvement resulted, that alone is not the measure of inventive ingenuity (*Asseff* v. *Kingsland; Sonneborn Sons, Inc.* v. *Coe,* both *supra; Rembert et al.* v. *Coe,* 78 U. S. App. D. C. 51).

Appellants' primary contention anent this rejection is that it is improper to combine these prior art teachings on the ground that the disclosures of the patents are radically different (Br. 16). In addition appellants appear to rely upon their interpretation of language appearing in Cook et al. parent application (Br. 25, 26). However, while it might be questioned whether the statement quoted really means that a combination of a dithiophosphate and a detergent is inoperative, as contended, it is significant that McNab states that the conjoint use of the soap type detergent and thiophenolates gave superior results (page 3, col. 1, lines 2–12). Since McNab teaches the equivalency of the thiophenolates and the thiophosphates, it would appear evident that the thiophosphates there disclosed would function in the same way. Hence, irrespective of what Cook et al. may say, the prior teaching of McNab stands out as recommending that the metallic soap type detergent and the calcium salt of a dioctyl dithiophosphate conjointing used in a lubricating composition for Diesel engines would give superior results with respect to corrosion and sludge formation. Appellants merely followed that teaching and the teaching of Salzberg when they used the zinc salt instead of the calcium salt disclosed by McNab. Following in the path blazed by the prior art, in the manner suggested by the prior art does not exhibit that degree of creative ingenuity characterized by the term invention.

3. Indefiniteness

In addition to the holding of nonpatentability over the prior art, the tribunals of the Patent Office held that claims 16, 18, 20, and 21 were indefinite and unduly broad because of the recitation therein of "a separate oil-soluble organic corrosion inhibitor" (App. 18). Further, in the opening statement by appellee's counsel it was pointed out that the answer denied patentability because of indefiniteness in many of the claims (App. 31). Although the trial court stated that it deemed it unnecessary to pass upon these contentions (App. 22), appellee submits them for the Court's consideration, if necessary.

With respect to the objection to the expression defining the corrosion inhibitor, it should be noted that the only indication of what the composition or constitution of the material so functioning is, is that it is organic. Reference to the application upon which the claims are based (App. 125, 126) shows that the claims are much broader than the application disclosure. While appellants urge that the expression is commonly used in the art and that Asseff's invention was not concerned with the combination with specific corrosion inhibitors (Br. 2), it is noted that the application itself does not state that any corrosion inhibitor can be used. Only "halogen bearing organic compounds" (App. 125) or the specific phenols and amines listed (App. 126) are disclosed as effective for this purpose in combination with the zinc salt of dicapryl dithiophosphate. Apart from the fact that the claim is clearly much broader than the disclosure upon which it is allegedly based it is desired to point out that the courts have condemned the description of a compound or composition solely in terms of function, and have held that some designation of ingredients is necessary for proper definition. (*Holland Furn. Co.* v. *Perkins Glue Co.*, 277 U. S. 245.) Defining the ingredient merely as a corrosion inhibitor does not tell the public what it is but only what it does in use, and what it does can be determined only by extensive experimentation. And where extensive experimentation must be resorted to to determine the metes and bounds of the patent monopoly, the claims are unduly broad and fall squarely within the field of prohibited indefiniteness. (*Standard Brands* v. *Nat. Yeast,* 308 U. S. 304.)

Claims 1, 9, 10, 11, 15 to 18, inclusive, and 21 to 24, inclusive, purport to define a lubricating composition, but actually what is defined therein will not operate as a lubricating composition for the intended purpose or use. The composition defined is only the additive. As defined the claims are indefinite and incomplete since they omit or include by implication only, if they include it at all, the major ingredient of the lubricating composition, i. e., the mineral lubricating oil. That element of the composition is essential to its functioning as a lubricant particularly where high temperature conditions are encountered. Appellants contend only that in commercial practice it is common to sell concentrates of the additives and that allowance of claims of this type are necessary for full protection (Br. 18). However, since the claims define a lubricating composition they should be complete, and where the incompleteness is due to the omission of an essential element, invalidity is the rule (*Brush* v. *Lexington,* 14 F. 2d 108; *Crampton Mfg. Co.* v. *Crampton,* 68 USPQ 122.) Moreover, in connection with appellants' contention it is desired to direct attention to the fact that other claims, such as 12, 13, 14 19, and 20, would amply protect commercial practice, if allowed, because they define the "addition agent."

Many of the claims (1, 9, 10, 11, 15 to 18, and 21 to 24) define the composition as containing "a characterizing amount" of the zinc dicapryl dithiophosphate. What is a characterizing amount? Appellants contend that the specification of the Asseff application is quite specific as to the percentages which may be used to give satisfactory results, and that the claims should be interpreted in the light of the specification (Br. 18). However, it is submitted that the claims must be read separately from the specification, and that the claims must particularly and distinctly point out the alleged invention (*Graver Tank et al.* v. *Linde Air Products,* 80 USPQ 451). Since the claim on its face suggests that any amount cannot be used to accomplish the results but only "characterizing amounts" it would appear that that term is intended to mean an amount sufficient to accomplish the desired result. If so, it is submitted that it is bad for indefiniteness under *Murphy & Chadeloid Chem. Co.* v. *Coe,* 69 App. D. C. 225, and that the expression does not meet the requirement in Sec. 4888 R. S. for definiteness.

In conclusion it is desired to point out that all the claims here involved have been found to be lacking in invention by two tribunals of the Patent Office and by the court below. The question as to whether or not a particular procedure would be obvious and within the skill of the art is a technical one on which the tribunals of the Patent Office are peculiarly adapted to pass. Where the conclusion of the Patent Office is further fortified by the decision of the District Court, the rule is well established that the finding of nonpatentability below will not be disturbed if it is a reasonable one in view of the evidence (*Abbott* v. *Coe,* 71 App. D. C. 195, 109 F. 2d 449; *Radtke et al.* v. *Coe,* 74 App. D. C. 251, 122 F. 2d 937). In other words, this Court will not substitute its opinion for that of the concurring tribunals below unless it is very clearly shown to be erroneous (*Standard Oil* v. *Marzall,* 84 USPQ 363).

CONCLUSION

It is respectfully submitted that in the present case appellants have failed to sustain the burden cast upon them in their attempt to antedate the anticipatory effect of the Cook et al. patent, and hence that the holding of the tribunals of the Patent Office and the District Court that all the claims are unpatentable over Cook et al. is sound; that the holding of the tribunals of the Patent Office and the District Court that all the claims are unpatentable over McNab, Wilson and Salzberg is sound; that claims 1, 9–11, 15–18 and 21–24 are unpatentable because they fail to meet in one or more particulars the statutory requirement for particularity and distinctness; and that the decision appealed from should be affirmed.

Respectfully submitted.

E. L. Reynolds,
Solicitor, U. S. Patent Office,
Attorney for Appellee.

J. Schimmel,
Of Counsel.

April 1950.

U. S. GOVERNMENT PRINTING OFFICE: 1950

APPENDIX B.

ASSEFF'S ACTUAL WORK.	SECRET DISCLOSURES.	PUBLIC DISCLOSURES.
		December 8, 1936 Salzberg patent 2,063,629 disclosing the acid esters and their salts of thioacids of phosphorus containing at least 1 aliphatic chain of at least 10 carbon atoms as new chemical compounds which are stated to be useful "as assistants for use in extreme pressure lubricants." No percentages were given and no data with regard to their performance was given.
	September 1, 1939 McNab filed a patent application in which he suggested that for use in lubricants "certain mixed soap compositions are very much superior to compositions containing a single soap." Included in the vast list of soaps was calcium dioctyl dithio phosphate. McNab gave no examples utilizing that particular soap. The record shows that that soap is virtually insoluble in oil.	
April-May 1940 Asseff ran engine tests on lubricants containing in combination certain detergents with certain salts of P_2S_5 treated alcohols. (Exhs. 47-48.)		
May 1940 Asseff ran an engine test on a lubricant containing the zinc salt of P_2S_5 treated normal octyl alcohol. (Ex. 49.)		
	July 16, 1940 Wilson filed a patent application suggesting the combined use in a lubricant of an oil soluble petroleum sulphonate (detergent) and a salt of a weak non-carboxylic organic acidic material having an ionization constant not exceeding about 5 x 10^{-6}. Wilson disclosed no thiophosphates.	
August 1940 Asseff ran engine tests on a lubricant containing a calcium petroleum sulphonate in combination with zinc salt of P_2S_5 treated methyl cyclohexanol. (Ex. 51.)		
	July 11, 1941 Cook et al. filed application 401,960 in which they stated "the use of a corrosive inhibitive in admixture with the detergents heretofore employed has proven to be ineffective over any extensive period of time as each chemical tends to offset the action of the other."	
	July 28, 1941 Asseff filed a patent application 404,361 giving a number of specific examples of lubricants containing various detergents in combination with the zinc salt of the acidic reaction product of P_2S_5 and an alkalated cyclohexanol.	
September 11, 1941 Asseff prepared the composition and October 3, 1941 completed full scale engine tests on a lubricant containing the combination of a detergent and the zinc salt of P_2S_5 treated capryl alcohol.		
		October 28, 1941 Asseff issued patent 2,261,047 disclosing the specific composition of 11 finished lubricants containing combined therein various detergents and the zinc salt of P_2S_5 treated cycloaliphatic alcohols.
	November 8, 1941 Cook et al. filed their application serial no. 418,326 disclosing the combined use in a lubricant of a detergent and a heavy metal salt of the dicapryl ester of di-thio phosphoric acid.	
		April 21, 1942 Wilson issued patent 2,280,419 based on his application filed July 16, 1940.
		July 17, 1942 McNab issued patent 2,289,795 based on his application filed September 1, 1939.
		March 14, 1944 Cook et al. issued patent 2,344,392 based on their application filed November 8, 1941.